3M Center St. Paul, MN 55144-1000

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May 30, 2000

2000 JUN -1 AN 71 69



Dr. Charles Auer Director **Chemical Control Division** Office of Pollution Prevention and Toxics United States Environmental Protection Agency 401 M Street, Southwest Room 403 East Tower (Mail Code 7405) Washington, D.C. 20460



Re:

Information of Perfluorooctane Sulfonates And Related Compounds – UEIP Forms



Dear Charlie:

3M is enclosing the completed "Use and Exposure Information Profiles" or "UEIPs" for compounds related to perfluorooctane sulfonates. As explained in my cover letter to the May 18th submittal to EPA, we needed additional time to clarify industrial hygiene and other release information to ensure that the data in the profiles is accurate and placed in appropriate context.

The enclosed information covers certain compounds related to perfluorooctane sulfonate as listed in Table 1 of the document entitled: "Sulfonated Perfluorochemicals in the Environment: Sources, Dispersion, Fate and Effects", at 12 (March 1, 2000). Specifically the UEIPs provided cover the following chemicals:

- *Perfluorooctanesulfonyl fluoride
- *Perfluorooctanesulfonamide
- *Perfluorooctane sulfonylamido (ethyl) acetate
- *Perfluorodecanesulfonate
- *N-ethyl perfluorooctanesulfonamide
- *N-methyl perfluorooctanesulfonamide
- *N-ethylperfluorooctane sulfonamidoethanol
- *N-methylperfluorooctane sulfonamidoethanol
- *N-ethylperfluorooctanesulfonamidoethyl acrylate
- *N-ethylperfluorooctanesulfonamidoethyl methacrylate
- *N-methyl perfluorooctanesulfonamidoethyl acrylate

A UEIP is not provided for one of the compounds, , perfluorohexane sulfonate, listed in Table 1 because it is not an octyl sulfonate.

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Page 2 Dr. Charles Auer

With respect to industrial hygiene information, you should be aware that each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

In certain situations, 3M has gathered personal industrial hygiene monitoring data for the specific compounds and have included that data in the profile. In some situations, there is no chemical-specific personal industrial hygiene monitoring data, but area/source monitoring data and/or surface wipe sampling data for the specific material may be available at the facility. These area/source sample results and/or surface wipe sample results are used to target areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. In other situations, neither personal sampling data nor area/source data have been collected for a specific chemical.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Similar to the industrial hygiene program, each 3M facility that produces sulfonated fluorochemicals has one or more environmental engineers on-site and is also supported by a corporate and division level environmental organization. The environmental engineers assist the process engineers with calculations and emission estimates. The corporate environmental organization develops methods for analyzing emissions. In addition to these resources, several teams were established in recent years to identify, characterize and reduce specific fluorochemical emissions from the manufacturing sites.

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process system emissions are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps.

Page 2

Dr. Charles Auer

Please do not hesitate to contact me at 651-733-6374 should you have any questions.

Very truly yours,

Hilliam a. Meppner William A. Weppner, Ph.D.

Director

Environmental, Health, Safety & Regulatory Affairs

Specialty Material Markets Group

3M Center, Building 236-1B-10

St. Paul, MN 55144-1000

Attachments

1

Voluntary Use and Exposure Information Profile Perfluorooctanesulfonyl fluoride (POSF)

CHEMICAL IDENTIFICATION I.

Chemical Name:

Perfluorooctanesulfonyl fluoride (POSF)

CAS Number:

307-35-7

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

22614 Route 84 North

Cordova, IL 61242

10746 Innovation Road Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
307-35-7*	> 1MM & < 10MM lbs.	0

Estimate the amount of subject chemical distributed off-site: 10% Exported

^{*}Submitted in 1998 TSCA IUR.

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluoroctanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

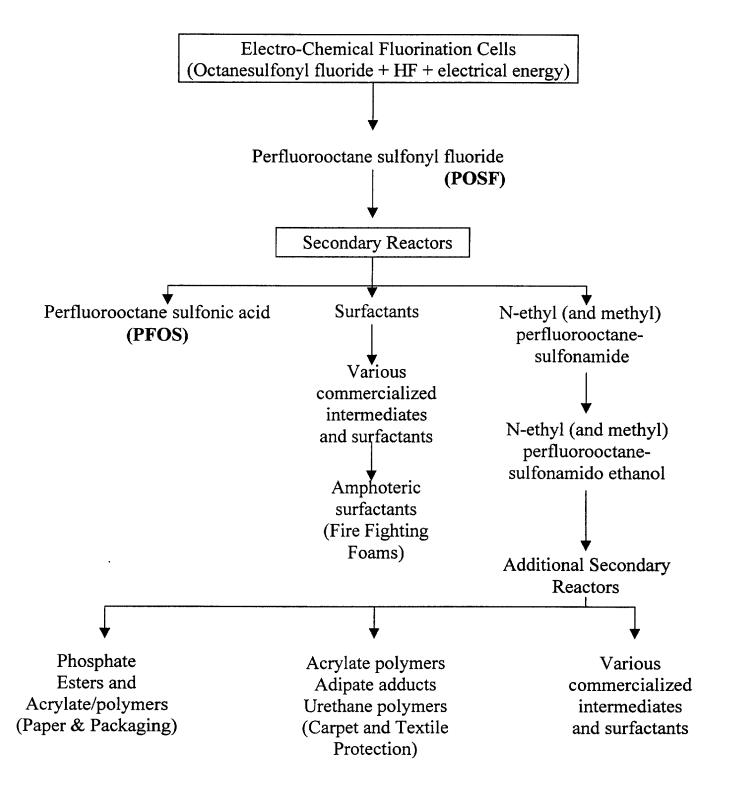
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS – Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material.

Industrial Hygiene monitoring has been conducted for some compounds and is detailed later in this document. Some minor amounts of these compounds have been detected as fugitive emissions during Industrial hygiene exposure testing, but do not contribute significantly to overall site emissions.

DECATUR, ALABAMA ONLY: Fugitive emission data is available for electrochemical cell buildings, wastewater fugitive air emissions, and fugitive emissions from wastewater sludge. Data for the cell buildings is based on engineering calculations using standard emission factors. Wastewater fugitive data is based on test results which show the presence of POSF. Sludge emission data is based on engineering calculations of the amount of POSF in the sludge. The C8 sulfonyl compound fugitive air emissions have been significantly reduced since 1997 as a result of reduction of wastewater discharges of materials containing these compounds. Additional reductions are planned.

Decatur, AL

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – (non-point)	<28,000 in 1997		250
	< 1700 in 1999		

Stack (point)

Engineering calculations and models of process vent emissions are used for estimates of point source emissions.

Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs	
5000		250	

Cordova

POSF-based products are no longer manufactured in Cordova. Stack point emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive (non-point)	No data available.		
Stack (point)	<1		<10 in 1997

Comments:

Cottage Grove

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive (non-point)	<1		< 100
Stack (point)	<1		< 100

B. WATER RELEASES FROM SITE

Decatur, AL

The data presented is part of wastewater testing conducted in 1998, since the individual compounds of interest were not analyzed in the 1997 wastewater material balance due to method availability and initial program focus. The 1998 data has not been adjusted for production levels in 1997, since most production remained at similar levels. POSF was measured on an infrequent basis because of analytical difficulties. When it was analyzed in wastewater effluent, it was not detected.

Estimated Total Annual Releases (lbs.) (1997)

Estimated %
Accuracy of
Estimate (optional)

Water releases:

0 in 1999

Number of days/year release occurs: 250

Receiving Water Name: Baker's Creek at the junction with the Tennessee River

NPDES Number: ALD004023164

Comments:

Cordova, IL

Engineering calculations were used to estimate the amount of material discharged to wastewater. The amount of material discharged to the river was determined through use of existing removal efficiency testing results from another facility.

Estimated Total Annual Releases (lbs.) (1997) Estimated %
Accuracy of
Estimate (optional)

Water releases:

< 10

Number of days/year release occurs: <5 times in 1997. POSF-based products no longer produced in Cordova.

Receiving Water Name: Mississippi River

NPDES Number: IL0003140

Cottage Grove, MN

Engineering calculations were used to estimate the amount of material discharged to wastewater. The amount of material discharged to the river was determined through use of existing removal efficiency testing results from another facility.

Estimated Total Estimated %
Annual Releases (lbs.) (1997) Estimate (optional)

Water releases:

0

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

Comments:

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation.

Levels of POSF in the sludge were estimated from engineering calculations.

Estimated Total Estimated %
Annual Releases (lbs.) (1997) Estimate (optional)

Landfill

Land Treatment/Land Amendment

Surface Impoundments Underground Injection

Other (specify):

0

26,000 - No longer used

No data available/No longer used

0

Cordova, IL

Less than 10 pounds total of all compounds per year are released to wastewater treatment (see wastewater section) from process. The amount of material discharged to land treatment was determined through the use of testing results from another facility.

The sludge formed in the primary clarifier is most likely to contain the insoluble sulfonated materials. This sludge is removed and incinerated. The sludge impoundments are lined.

The use of POSF containing materials has been discontinued at Cordova so these releases have also been discontinued.

	Estimated Total Annual Releases	Estimated % Accuracy of
	(lbs.) (1997)	Estimate (optional)
Landfill	0	
Land Treatment/Land Amendmen	t < 10	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):	0	

Comments:

Cottage Grove, MN

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

Estimated Total Annual Releases	Estimated % Accuracy of	
(lbs.) (1997)	Estimate (optional)	
0		
0		
0		
0		
	Annual Releases (lbs.) (1997) 0 0 0	

D. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Comments:

Cordova, IL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather, wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

A review of plant records regarding waste disposal locations for Decatur fluoride-containing (not CAS number specific) wastes indicates that 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the primary disposal method for these materials.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS nu	mber data available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	No specific CAS nu	mber data available.
Other Landfill	No specific CAS nu	mber data available
Recycle or Recovery	0	
Unknown or Other	0	
· Comments:		

Cordova, IL

Cordova wastes that contained POSF were all incinerated. Wastes included some off-spec product, empty bags and empty drums and contained the compounds is residual amounts. These products are no longer made at Cordova.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS nu	mber data available
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other	0	
Comments:		

14

Cottage Grove, MN

Comments:

Cottage Grove facility utilizes incineration for all their drummed wastes.

Sludge from the Cottage Grove facility is sent to an industrial landfill.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	6490	
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other		

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Cordova, IL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<.25				
.25-1	1			
1-8	5			
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

The chemical (307-51-7) is produced as a by-product in the electrochemical fluorination process for 307-51-7. Employees take quality samples during the fluorination process. The liquid product containing 3% 307-35-7 is piped to a process vessel where it is washed and then drained into drums. The washed, drummed material is shipped to 3M Cottage Grove. Employees also perform turnaround maintenance on fluorination equipment.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Local exhaust ventilation is used at the drum tops during draining and sampling. Both room dilution ventilation and local exhaust ventilation is scrubbed. Production and work-up rooms are negatively pressured with respect to other parts of the building.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

During sampling, fluorination equipment turnaround, and drumming tasks, employees wear supplied air-fed fully encapsulating suits with attached butyl gloves and rubber boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<.25	14	16		
.25-1	14	16		
1-8	20	8		
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

The chemical (307-35-7) is a clear liquid. The chemical is handled as a raw ingredient in its pure form (95-100%), and as a component in other raw materials at concentrations typically < 5%. After initial processing of raw materials containing 307-35-7, the chemical may be carried over as a trace material (typically < 1%) into a variety of other materials during subsequent manufacturing steps. Workers may be exposed to 307-35-7 during collection of quality samples from drums, vacuum charging the material from drums into reactors, or during draining of materials from reactors into drums or other containers.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program is based on the concept of task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Local exhaust ventilation is used at drum top and reactor openings during sampling, charging, and draining activities. Vacuum charging of materials from drums is a standard practice. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

During sampling and charging of pure (95%) 307-35-7, workers use full facepiece air-purifying respirators with organic vapor/acid gas cartridges, rubber gloves (butyl or neoprene), and a two-piece or one-piece full body acid suits. The type of personal protective equipment used may vary (may be downgraded) for materials containing 307-35-7 as a trace ingredient; exact PPE used depends on the hazard posed by the main ingredient and specifications of the process operating standard.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<0.25				
0.25-1	12		24	
1-8	21	32	12	
>8	4		8	

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Operating fluorination cell system (closed), sampling, drumming, charging, and equipment maintenance activities (change pipe, flange, valve, filter, pump or sight glass); perfluorooctanesulfonyl fluoride is handled as a liquid; exposure is by inhalation or direct skin contact; material concentration ranges up to 80%.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; perfluorooctanesulfonyl fluoride exposure is typically to vapors; concentration in waste stream typically less than 2%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

Personal sampling for perfluorooctanesulfonyl fluoride vapor is currently (late 1999 to present) done with evacuated canisters with analysis by GC-MS. Previous sampling had employed silica gel acid mist tubes and XAD-4 tubes with GC-ECD analysis. Perfluorooctanesulfonyl fluoride was found to hydrolyze on sorption tubes resulting in an underestimate of exposure. See attached table for air sample results.

There has been area/source air monitoring data and/or surface wipe sampling data collected for this material at the plant. Area/source sample results and/or surface wipe sample results are used to identify areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. Prior to 1999, these samples were considered to be semi-validated.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds, including compounds covered in this submittal. The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

Air Sampling Data for 307-35-7

Job Classification	Sample number	Concentration (mg/m3)	Minimum (mg/m3)	Maximum (mg/m3)	Geometric Mean	Geometric Standard Deviation
		Totals →	<0.07	34.4	0.4	7.5
Chemical	95-042*	0.24				
Operator						
Building 3						
	96-028*	0.43				
	96-053*	0.07				
	96-067*	0.1				
	96-071*	1.12				
	97-124	0.12				
Team Leader	98-098*	0.18			· · · · · · · · · · · · · · · · · · ·	-
Building 3						
	98-481*	34.436				

^{*} The value for this sample is one of several fluorochemical analytes collected on the sample. Samples are all personal breathing zone samples taken over the duration of a specific task.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Area surveys using FTIR are performed to identify sources such as system leaks. Perfluorooctanesulfonyl fluoride is transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of perfluorooctanesulfonyl fluoride or perfluorooctanesulfonyl fluoride-containing materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. Some drumming operations are done in an enclosed ventilation chamber. General room air provides for dilution of airborne perfluorooctanesulfonyl fluoride. Operator control rooms are segregated from process areas and provided separate room air ventilation.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Neoprene suits (primarily for hydrogen fluoride exposure potential) are used in fluorochemical cell areas for tasks identified as having exposure potential. Exposure significant tasks in other areas use one or two-piece PVC suits.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

Current exposures are less than those measured in the recent past. Significant sources have been identified and eliminated. In 1998 an extensive area air sampling of the production areas was performed. Areas with higher levels of perfluorooctanesulfonyl fluoride were investigated further using FTIR to identify sources of perfluorooctanesulfonyl fluoride. Eliminated sources include a leaking pipe flange in a process area and vapors from an open chemical sewer. In addition, the ventilation system in the primary production building (3) is in the process of being upgraded.

The fluorochemical hazard communication program was improved upon in 1998. This has resulted in improved exposure avoidance behaviors among production employees and better use of protective equipment requirements.

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

(Inclu	Product chemical class or product chemical de CAS number if appropriate)	% of total volume of subject chemical manufactured or imported
1.	Perfluorooctanesulfonyl fluorides (POSF) are used as an intermediate to make consumer and commercial upholstery and carpet protectors and cleaners.	2%
2.	Perfluorooctane sulfonylamido ethyl acetate CAS #2991-51-7 POSF is used as an intermediate to make a household cleaner.	< 0.5%
3.	N-methylperfluorooctylsulfonamide CAS #31506-32-8 POSF is used as an intermediate to make polymers in protective treatments for nonwoven materials.	< 1.0%
4.	N-methylperfluorooctylsulfonamido ethanol CAS #24448-09-7 POSF is used as an intermediate to make polymers in protective treatments for Carpet, upholstery and leather materials.	36%
5.	N-methylperfluorooctanesulfonamido ethyl acrylate CAS #25268-77-3 POSF is used as an intermediate to make Consumer carpet protectors.	< 1.0%
6.	N-ethylperfluorooctylsulfonamido ethanol CAS #1691-99-2 POSF is used as an intermediate to make polymers in protective treatments for carpet, upholstery, apparel and leather products. It is also consumed in products manufactured for surfactants and coating additives.	48%

7. N-ethylperfluorooctylsulfonamide
CAS #4151-50-2
POSF is an intermediate consumed in the
Production of surfactants, powder additives,
Foam tape and insecticide.

< 1.0%

8. Fluorinated alkyl amine oxide CAS #178094-69-4
POSF intermediate consumed in the production of foamer for use in fire fighting foams.

< 1.0%

9. Fluoroalkyl Quaternary Ammonium CAS #38006-74-5
POSF intermediate consumed in the production of surfactants for the mining and oil well industries.

< 1.0%

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Exported.

< 10%

As reported in Part III, p.2

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

Use Number 1 of 1

Description of Chemical End Use: Used as a reactive intermediate in the synthesis of a foamer for use in fire fighting foams. These products are designed for primary use by fire fighting professionals. CAS 307-35-7 would be present in the final product as a low level residual.

Percent of total manufactured or imported volume going to this use: $\leq 1 \pm 1$	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid
X <1% 600 – 1200 ppm) 1-30% 30-60% 60-90% >90%	Gas or vapor X Liquid solution Other (Explain)

1

Voluntary Use and Exposure Information Profile Perfluorooctane sulfonamide

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

Perfluorooctane sulfonamide

CAS Number:

754-91-6

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
754-91-6	0 No Commercial Activity	0

Estimate the amount of subject chemical distributed off-site:

% of manufacture/import

If you have already provided the above information to EPA and it is still representative please reference that submission here:

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluoroctanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

$$\begin{array}{c} Energy \\ C_8H_{17}S0_2F \ + \ 17\ HF \\ \hline ECF \\ \hline 1-Octanesulfonyl\ fluoride \\ \end{array}$$

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

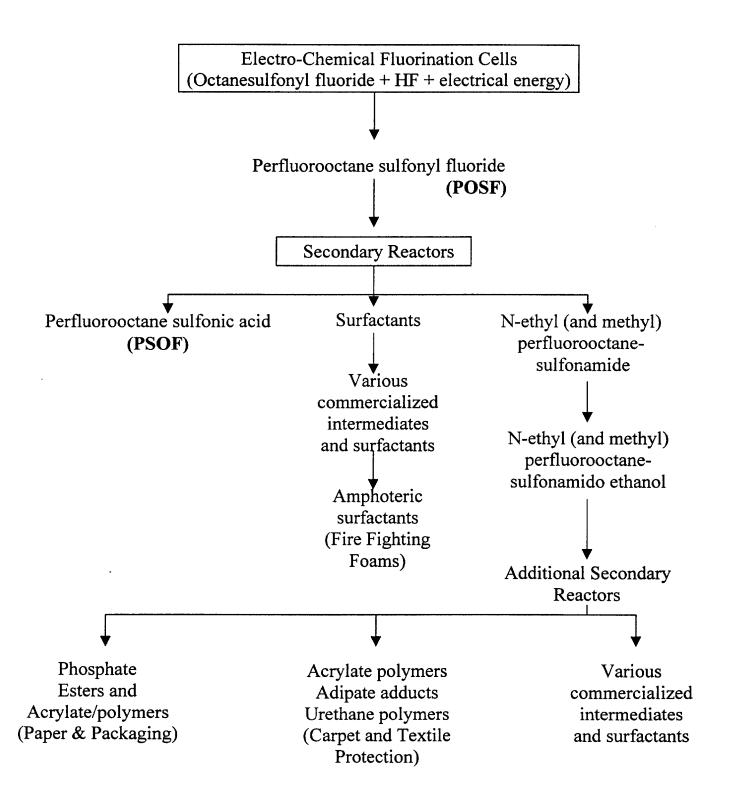
Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

- ------

CAS number 754-91-6, perfluorooctanesulfonamide (R-SO₂NH₂), is a small undesired byproduct of the reaction of POSF with methyl or ethylamine because of the presence of a small amount of ammonia (NH₃) in the methyl or ethylamine raw material. Perfluorooctane sulfonamide is not produced for a commercial purpose. This chemical is present in very small amounts (0.1% or less) in the various POSF based intermediates produced by 3M. It can theoretically be present in final 3M products but is currently monitored only in 3M FDA regulated indirect food additive products. It is present at approximately 0.01% in the latter products.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS - Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

Estimated Total Annual Releases (lbs.) Estimated %
Accuracy of
Estimate (optional)

#days/years release occurs

Fugitive (non-point)

No data available.

Stack (point)

No data available.

WATER RELEASES FROM SITE В.

Decatur, AL

The data presented is part of wastewater testing conducted in 1998, since the individual compounds of interest were not analyzed in the 1997 wastewater material balance due to method availability and initial program focus. The 1998 data has not been adjusted for production levels in 1997, since most production remained at similar levels.

Materials in the sludge and wastewater may result from production losses in the manufacturing of these compounds or from the hydrolysis of more complex molecules.

> **Estimated Total** Annual Releases (lbs.)

Estimated % Accuracy of Estimate (optional)

Water releases:

370

Number of days/year release occurs: 250 days/year

Receiving Water Name: Baker's Creek at Junction with Tennessee River

NPDES Number: ADL004023164

Comments:

Cottage Grove, MN

Estimated Total

Annual Releases

(lbs.)

Estimated %

Accuracy of Estimate (optional)

Water releases:

No data available.

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

Comments:

Cordova, IL

Estimated Total

Annual Releases (lbs.)

Estimated % Accuracy of

Estimate (optional)

Water releases:

No data available.

CAS Number 754-91-6

3M Company

7

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation. Levels of the compound in the sludge were determined from wastewater data.

Estimated Total	Estimated %
Annual Releases	Accuracy of
(lbs.) (1997)	Estimate (optional)

Landfill	No data available.	
Land Treatment/Land Amendment	30	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

Comments:

Cordova, IL

The manufacture of these compounds has been discontinued so these releases have also been discontinued.

Engineering calculations were used to estimate the amount of material discharged to wastewater. The amount of material discharged to the sludge was determined through use of existing removal efficiency testing results from another facility.

Estimated Total

Estimated 0/

	Annual Releases	Accuracy of	
	(lbs.) (1997)	Estimate (optional)	
Landfill	0		
Land Treatment/Land Amendmen	nt < 9		
Surface Impoundments	0		
Underground Injection	0		
Other (specify):			

Comments:

Cottage Grove, MN

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

	Estimated Total Annual Releases	Estimated % Accuracy of	
	(lbs.) (1997)	Estimate (optional)	
Landfill	0		
Land Treatment/Land Amendment	0		
Surface Impoundments	0		
Underground Injection Other (specify):	0		

Comments:

C. OFF-SITE TRANSFERS

D1. Transfer to Publicly Owned Treatment Works (POTW)

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

Number of days/year the release occurs:

Annual Transfer (lb):

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country: Zip Code:

State: NPDES Number:

Comments:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather, wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

CAS Number 754-91-6

3M Company

Estimated Total Estimated % Annual Releases Accuracy of

(lbs.)

Estimate (optional)

10

Incineration:

No specific CAS number data available.

Wastewater Treatment (Excluding POTW)

0

Underground Injection

0

Hazardous Waste (RCRA Subtitle C) landfill

No specific CAS number data available.

Other Landfill

No specific CAS number data available.

Recycle or Recovery

No specific CAS number data available.

Unknown or Other

Comments:

VI. ON-SITE WORKPLACE EXPOSURE

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

Hrs/day	Days/yr.			
	< 10	10-100	100-250	>250
<2.5				
25-1				
1-8				
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

This material is not handled at any of the facilities.

- 3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.
- 4. Briefly describe the engineering controls used to minimize exposure to this chemical:

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

COMMENTS:

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1.

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1.

As reported in Part III, p.2

Use Number	1 of 1		
Description	of Chemical	End	Use:

Percent of total manufactured or imported volume going to this use: ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: <1 1-30% 30-60% 60-90% >90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)

1

Voluntary Use and Exposure Information Profile Perfluorooctane sulfonamido ethyl acetate

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

Perfluorooctane sulfonamido ethyl acetate (PFOSSA)

CAS Number:

2991-51-7

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

1400 State Docks Road

Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
2991-51-5*	< 100,000 lbs.	0

Estimate the amount of subject chemical distributed off-site:

93.5% of manufacture/import

*Submitted in 1998 TSCA IUR.

If you have already provided the above information to EPA and it is still representative please reference that submission here:

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluoroctanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

Energy
$$C_8H_{17}SO_2F + 17 HF$$
 $C_8F_{17}SO_2F + 17 H_2$
ECF

1-Octanesulfonyl fluoride

Perfluorooctanesulfonyl fluoride

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

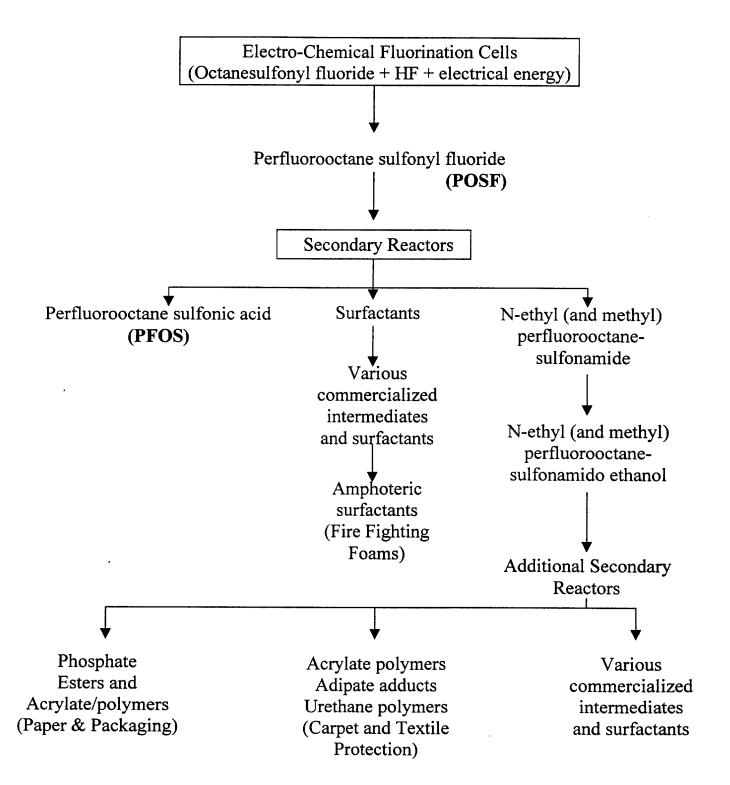
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS - Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

DECATUR, ALABAMA ONLY:

Decatur, AL

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

been quantified.			
	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – (non-point)	No data available.		
Stack (point)			
	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs

B. WATER RELEASES FROM SITE

Decatur, AL

The data presented is part of wastewater testing conducted in 1998, since the individual compounds of interest were not analyzed in the 1997 wastewater material balance due to method availability and initial program focus. The 1998 data has not been adjusted for production levels in 1997, since most production remained at similar levels.

It is not possible to distinguish the amount of chemicals generated from specific reaction steps in production of various compounds, or whether their presence may be the result of hydrolysis of more complex molecules.

Estimated Total Estimated %
Annual Releases (lbs.) (1997) Estimate (optional)

Water releases:

420

Number of days/year release occurs: Most compounds are manufactured on a regular basis, so releases are estimated at 250 days per year.

Receiving Water Name: Baker's Creek at the junction with the Tennessee River

NPDES Number: ALD004023164

Comments:

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation. Levels of the compound in the sludge were determined from wastewater data.

Estimated Total Estimated %
Annual Releases Accuracy of
(lbs.) (1997) Estimate (optional)

Landfill

0

Land Treatment/Land Amendment

8.100

No longer used

Surface Impoundments

No data available/No longer used

Underground Injection

0

Other (specify):

Comments:

D. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Comments:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the predominant disposal method for these materials.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS nu	mber data available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	No specific CAS nu	mber data available.
Other Landfill	No specific CAS nu	mber data available.
Recycle or Recovery	0	
Unknown or Other		
Comments:		

9

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<0.25				
0.25-1	11			
1-8	16	24		
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

The chemical (2991-51-7) is sampled and drained into drums in a molten state at 80-90% concentration. The material is vacuum-charged in a molten state from drums and formulated into a liquid at 42% concentration. The process mixture is sampled and the final solution is drummed at room temperature. Additional exposure may also occur during equipment maintenance.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Local exhaust is used during drumming and charging of molten material, and during drumming of the formulated solution.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Not Applicable

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Exported

3%

As reported in Part III, p.2

Use Number 1 of 3

>90%

Description of Chemical End Use: Chemical formulators use this chemical as a surfactant to improve the wetting of water-based products that are sold as cleaners.

Percent of total manufactured or imported volume going to this use: 66 ± 1	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: X <1% (50-100 ppm) 1-30% 30-60% 60-90% >90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)
Use Number 2 of 3 Description of Chemical End Use: Chemical form to improve the wetting of water-based products that	
Percent of total manufactured or imported volume going to this use: 29± 1	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid
X <1% (50-100 ppm)	Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)

Use Number 3 of 3

Description of Chemical End Use: Formulators use this chemical as a surfactant in a personal care consumer denture cleaner.

	t of total manufactured or imported going to this use: 3 ± 0.25	k all physical forms of the ical during this use:
to indic	in a mixture check appropriate box cate weight fraction. Average are acceptable: <1% 1-30% 30-60%	 Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)
	60-90% >90%	

1

Voluntary Use and Exposure Information Profile Perfluorodecane sulfonate

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

Perfluorodecane sulfonate (PDSF)

CAS Number:

335-77-3 (acid)

67906-42-7 (ammonium salt)

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

<u>CAS #</u> *	Mfg. (1997)	<u>Imported</u>
335-77-3	0	0
67906-42-7	0	0

Estimate the amount of subject chemical distributed off-site:

% of manufacture/import

If you have already provided the above information to EPA and it is still representative please reference that submission here:

^{*}These materials are not produced during POSF (CAS #307-35-7) manufacture. Manufacturing did not occur in 1997 but did occur in 1998 and 1999 at < 100,000 lbs.

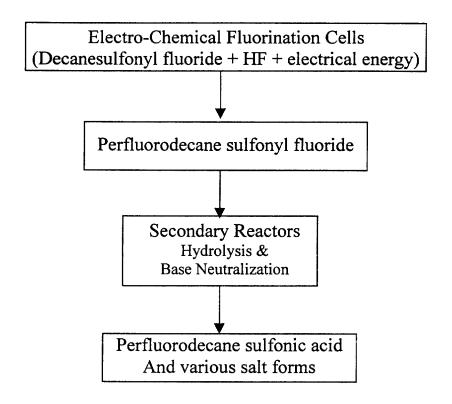
Narrative Description and Process Flow Schematic:

Perfluorodecane sulfonates are synthesized via the Simons Electro-Chemical Fluorination (ECF) process. The starting feedstock for this reaction is Decane Sufonyl Fluoride

PDSF is not itself a commercially viable product, but is used as an intermediate in the manufacture for Perfluorodecane sulfonates. The perfluorodecane sulfonic acid is manufactured by hydrolyzing perfluorodecane sulfonyl fluoride to the corresponding sulfonic acid in batch reactors. The salts are manufactured by base neutralization of the acid to the salt in a separate reactor. The low sales volume and reactor availability dictate an infrequent production schedule for these chemicals.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

Cottage Grove, MN

Engineering calculations and models of process vent emissions are used for estimates. Existing process emission information shows only minor losses through point source vents.

Fugitive emissions may have occurred during some handling steps but have not been quantified.

Estimated Total Annual Releases (lbs.)

Estimated %
Accuracy of
Estimate (optional)

days/years release occurs

No data available.

Stack (point)

Fugitive (non-point)

< 1

Comments:

B. WATER RELEASES FROM SITE

Cottage Grove, MN

1000 pounds of PDSF were estimated discharged to the wastewater treatment system in 1998. No data is available for 1997. The level of this compound discharged, based upon behavior of POSF in Decatur, is believed to be close to zero (<1% of total discharge).

Continued improvements in reducing material to the river are being undertaken by a wastewater emission reduction team and engineers at the facility.

59

Estimated Total

Estimated %

Annual Releases

Accuracy of Estimate (optional)

Water releases:

< 10

(lbs.)

Number of days/year release occurs: 6/year

Receiving Water Name: Mississippi

NPDES Number: MN00001449

Comments:

C. ON-SITE LAND RELEASES

Cottage Grove, MN

Estimated Total	Estimated %
Annual Releases	Accuracy of
(lbs.)	Estimate (optional)

Landfill 0
Land Treatment/Land Amendment 0
Surface Impoundments 0
Underground Injection 0
Other (specify):

Comments:

D. OFF-SITE TRANSFERS

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City: Country: State: Zip Code:

NPDES Number:

Comments:



D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather, wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included. Incineration is now the primary disposal method for these wastes.

Cottage Grove, MN

Comments:

Cottage Grove facility utilizes incineration for all their drummed wastes.

Sludge from the Cottage Grove facility is sent to an industrial landfill.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	2,100	
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other		

VI. ON-SITE WORKPLACE EXPOSURE (335-77-3)

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<.25	4	8		
.25-1	4	8		
1-8	4	4		
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

The chemical (335-77-3) is a produced in an aqueous slurry, drained into drums. The material is vacuum charged as an aqueous slurry to produce other materials. Employees take quality samples during the process. The liquid product contains 33-43% 335-77-3.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Local exhaust ventilation is used at the drum tops during charging and drumming.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

During sampling, employees wear full faceshields, rubber suits and boots, and PVC gloves. During charging and draining employees wear either full face supplied air respirators or full facepiece OVAG air-purifying respirators, butyl gloves, and rubber jackets.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

VI. ON-SITE WORKPLACE EXPOSURE (67906-42-7)

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<.25	4	8		
.25-1	4	8		
1-8	4	4		
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

The chemical (67906-42-7) is a produced in an alcohol-aqueous solution, drained into drums. Employees take quality samples during the process. The liquid product contains 25% 67906-42-7.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Local exhaust ventilation is used at the drum tops during charging and drumming.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

During sampling, employees wear full faceshields, rubber jackets and boots, and PVC gloves. During draining employees wear full facepiece OVAG air-purifying respirators, butyl gloves, and rubber jackets.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Perfluorodecane sulfonic acid (CAS #335-77-3) is used as an intermediate to make perfluorodecane sulfonate, ammonium salt (CAS #67906-42-7).

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Perfluorodecane sulfonate, ammonium salt

50

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Not Applicable

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

T	Ice	N	ıım	ber	1	αf	2
ι	750	1.1	ши	UCL		O1	_

Description of Chemical End Use: Chemical formulators use this chemical as a surfactant to improve wetting and leveling of products sold as floor polishes. Janitorial service firms are the end users.

Percent of total manufactured or imported volume going to this use: 10 ± 2	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: \[\frac{X}{2} < 1\% (0.01-0.02\%) \\ \frac{1-30\%}{30-60\%} \\ \frac{60-90\%}{20\%} \\ \frac{60-90\%}{20\%} \\ \frac{200\%}{20\%} \]	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)
Use Number 2 of 2 Description of Chemical End Use: Chemical form to improve wetting and leveling of products sold as the end users.	
Percent of total manufactured or imported volume going to this use: 40 ± 5	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)

Voluntary Use and Exposure Information Profile N-ethyl perfluorooctanesulfonamide

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

N-ethyl perfluorooctanesulfonamide (EtFOSA)

CAS Number:

4151-50-2

COMPANY IDENTIFICATION II.

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
4151-50-2*	< 100.000 lbs.	0

Estimate the amount of subject chemical distributed off-site:

59% of manufacture/import

If you have already provided the above information to EPA and it is still representative please reference that submission here:

^{*}Submitted in 1998 TSCA IUR. Significant production occurs that is non-isolated and nonreportable under TSCA.

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluoroctanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

$$\begin{array}{c} Energy \\ C_8H_{17}S0_2F \ + \ 17\ HF \\ \hline ECF \\ \hline 1-Octanesulfonyl\ fluoride \\ \end{array} \begin{array}{c} Energy \\ \hline ------> \\ ECF \\ \hline Perfluorooctanesulfonyl\ fluoride \\ \end{array}$$

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

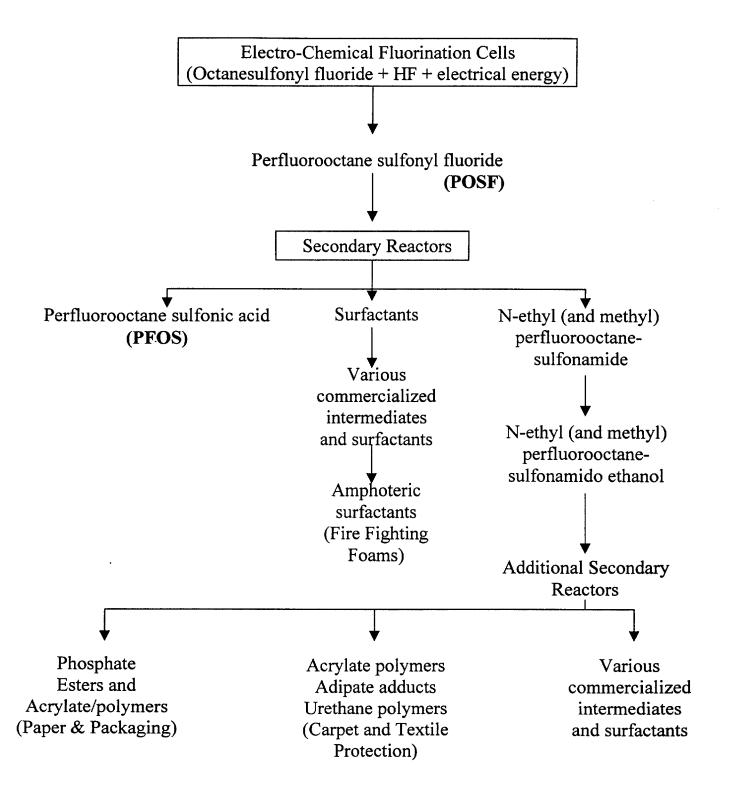
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS - Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

Decatur, AL

Wastewater fugitive emission data was based upon wastewater testing.

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

been quantified.	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – (non-point)	< 40 in 1997 < 40 in 1999		250
Stack (point)			
	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs

Cottage Grove

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive (non-point)	No data available.		<10
Stack (point)	No data available.		<10

B. WATER RELEASES FROM SITE

Decatur, AL

The data presented is part of wastewater testing conducted in 1998, since the individual compounds of interest were not analyzed in the 1997 wastewater material balance due to method availability and initial project focus. The 1998 data has not been adjusted for production levels in 1997, since most production remained at similar levels.

It is not possible to distinguish the amount of chemicals generated from specific reaction steps in production of various compounds, or whether their presence may be the result of hydrolysis of more complex molecules.

Estim	ated Total	Estimated %
Annu	al Releases	Accuracy of
(lbs.)	(1997)	Estimate (optional)

Water releases:

140

Number of days/year release occurs: 250

Receiving Water Name: Baker's Creek at the junction with the Tennessee River

NPDES Number: ALD004023164

Comments:

Cottage Grove, MN

Engineering calculations were used to estimate the amount of material discharged to wastewater. The amount of material discharged to the river was determined through use of existing removal efficiency testing results from another facility.

Estimated Total	Estimated %
Annual Releases	Accuracy of
(lbs.) (1997)	Estimate (optional)

Water releases:

-0

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation. Levels of the compound in the sludge were determined from wastewater data.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	
Landfill	0		
Land Treatment/Land Amend	ment 0		
Surface Impoundments	No data available/N	o longer used	
Underground Injection	0	· ·	
Other (specify):			

Comments:

Cottage Grove, MN

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

	Estimated Total Annual Releases	Estimated % Accuracy of
	(lbs.) (1997)	Estimate (optional)
Landfill	0	
Land Treatment/Land Amendment	0	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

C. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site releases cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the predominant disposal method for these materials.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS nu	mber data available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	No specific CAS nu	mber data available.

Other Landfill No specific CAS number data available.

Recycle or Recovery 0

Unknown or Other

Comments:

Cottage Grove, MN

Cottage Grove facility utilizes incineration for all their drummed wastes.

Sludge from the Cottage Grove facility is sent to an industrial landfill.

Estimated Total

Estimated %

	Annual Releases (lbs.)	Accuracy of Estimate (optional)
Incineration:	No specific CAS nu	ımber data available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other	0	
Comments:		

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

		Days	s/Year	
Hours/Day	<10	10-100	100-250	>250
<.25	6			
.25-1	2			
1-8	4			
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state or dissolved in organic solvents; vapors are produced from molten material; approximate concentration ranges from <1% to 80%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Positionable local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Process operating standards list the respirator (e.g., supplied air, organic vapor cartridge with particulate prefilter, or particulate filtering), glove by elastomer (e.g., neoprene or nitrile), chemical protective clothing (e.g., 2-piece PVC rainsuit), eye protection (e.g., chemical splash goggles with or without full faceshield depending on type of respirator used) to be used by the employee when the task involves exposure to a particular fluorochemical material.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<0.25				
0.25-1	4	4	24	
1-8		24		
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state; vapors are produced from molten material; material concentration ranges from <1% to 80%.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; materials typically present in waste stream at less than 1%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

Personal sampling for these materials is currently (late 1999 to present) done using OSHA Versatile Sampler tubes with XAD-4 resin and mixed cellulose ester or glass fiber prefilter. Sample analysis is by LC-MS. Previous sampling had employed silica gel acid mist tubes with GC-ECD analysis. See attached table for air sample results.

There has been area/source air monitoring data and/or surface wipe sampling data collected for this material at the plant. Area/source sample results and/or surface wipe sample results are used to identify areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. Prior to 1999, these samples were considered to be semi-validated.

Surfaces in production and administration areas were sampled in 1998. A glass fiber filter wetted with methyl alcohol was wiped inside a 10 cm x 10 cm square with analysis by LC-MS. Results indicated the presence of these materials on floors and equipment surfaces in production areas. This resulted in improvements to Hazard Communication practices, personal hygiene emphasis, personal protective equipment emphasis, and several engineering and administrative changes.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds, including compounds covered in this submittal. The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

Air Sampling Data for 4151-50-2

Job Classification	Sample number	Concentration (mg/m3)	Minimum (mg/m3)	Maximum(mg/m3)	Geometric Mean	Geometric Standard Deviation
		Totals →	<0.005	0.1	0.01	2.3
Chemical Operator	94-031*	0.015				
Building 3						
	94-032*	<0.01				
	94-034*	< 0.005				
	94-037*	0.004				
	94-038*	0.007				
	94-039*	0.013				
	94-041*	0.008				
	94-046*	0.007				
	94-050*	0.005				
Team Leader Building 3	94-036*	0.014				
•	94-047*	<0.02				
	94-051*	<0.005				
	94-054*	0.012				
	94-055*	0.1				

^{*} The value for this sample is one of several fluorochemical analytes collected on the sample. Samples are all personal breathing zone samples taken over the duration of a specific task.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials. Operator control rooms are segregated from process areas and provided separate room air ventilation.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

Current exposures are expected to be less than those measured in the recent past. Significant sources have been identified and eliminated. In 1998 an extensive area air sampling of the production areas was performed. Areas with higher levels of airborne materials were investigated further to identify sources. These sources, which include the flaking equipment, are in the process of being eliminated or reduced through replacement with better ventilated equipment. In addition, the ventilation system in the primary production building (3) is being upgraded.

The fluorochemical hazard communication program was improved upon in 1998. This has resulted in improved exposure avoidance behaviors among production employees and better use of personal protective equipment.

Additional hand wash facilities have been installed in primary process buildings. Food consumption and other hand-to-mouth activities are prohibited. There is no smoking in process buildings. Process employees are instructed to remove and leave their work boots and work coveralls at the plant. Work coveralls are professionally laundered. Non-production equipment surfaces such as floors, doorknobs and stair rails are cleaned frequently.

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Fluorinated alkyl sulfonamides are used as an intermediate in the production of fluorinated alkyl alkoxylates, and as an intermediate used to make foam tape additives.

Product chemical class or product chemical (Include CAS number if appropriate) % of total
volume of subject
chemical manufactured
or imported

1. Fluorinated CAS #68958-61-2 alkyl alkoxylate

75-80

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Not Applicable

Not Applicable

As reported in Part III, p.2

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

Use Number 1 o)Τ	0
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Description of Chemical End Use: Chemical formulators use this chemical as a registered active ingredient for insect control. It is blended with bait that is attractive to insects and placed in bait stations. Either commercial facilities or consumers can use these bait stations.

Percent of total manufactured or imported volume going to this use: 19 ± 1	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: X <1% (0.5-1.0%) 1-30% 30-60% 60-90% >90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution X Other (Explain) Waxy Solid
Use Number 2 of 6 Description of Chemical End Use: Chemical form to improve the wetting of protective coatings.	nulators add this chemical as a surfactant
Percent of total manufactured or imported volume going to this use: 2 ± 0.5	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid
X <1% (20 ppm) 1-30% 30-60% 60-90% >90%	Gas or vapor X Liquid solution Other (Explain)

Use Number 3 of 6

Description of Chemical End Use: Oil well service formulators and oil companies use this chemical as a surfactant in a well stimulation formula that is injected into an oil well to enhance the recovery of oil and gas.

Check all physical forms of the chemical during this use:
Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)
ations utilizing the solution mining process g of other chemicals that leach the metals
Check all physical forms of the chemical during this use:
Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vaporX Liquid solution Other (Explain)

Use Number 5 of 6

Description of Chemical End Use:	Powder additive formulators use this chemical as a
wetting agent for thickeners (Carbope	ol).

Percent of total manufactured or imported volume going to this use: < 1	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: \[\frac{X}{-} & <1\% \\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vaporX Liquid solution Other (Explain)
Use Number 6 of 6 Description of Chemical End Use: A formulator aid the gelling of medical waste solutions.	of powder additives adds this chemical to
Percent of total manufactured or imported volume going to this use: < 1	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid
X <1% 1-30% 30-60% 60-90% >90%	Gas or vapor X Liquid solution Other (Explain)

1

Voluntary Use and Exposure Information Profile N-Methyl perfluorooctanesulfonamide

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

N-Methyl perfluorooctanesulfonamide (MeFOSA)

CAS Number:

31506-32-8

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	Imported
31506-32-8*	< 100.000	0

Estimate the amount of subject chemical distributed off-site: 0

^{*}Submitted in 1998 TSCA IUR. Significant production occurs that is non-isolated and non-reportable under TSCA.

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluoroctanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

Energy
$$C_8H_{17}S0_2F + 17 HF$$
 $C_8F_{17}S0_2F + 17 H_2$
ECF

1-Octanesulfonyl fluoride

Perfluorooctanesulfonyl fluoride

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

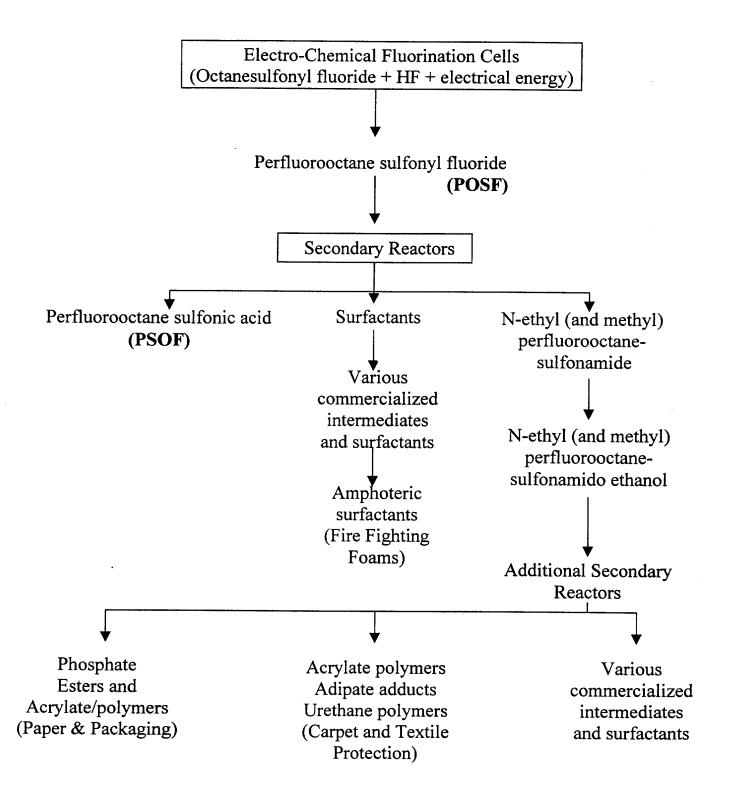
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS - Fugitive emission may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

DECATUR, ALABAMA ONLY:

Decatur, AL

Emission estimates are based upon existing models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

Wastewater fugitive emission data was based upon wastewater testing.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – wastewater			
	< 100 in 1997		250
	< 100 in 1999		
Stack (point)			
	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs

Cottage Grove

Emissions estimates are from process engineer's estimates and emission models.

Estimated Total Estimated % # days/years
Annual Releases Accuracy of release occurs
(lbs.) (1997) Estimate (optional)

Fugitive (non-point) No data available.

Stack (point) 0

B. WATER RELEASES FROM SITE

Decatur, AL

This compound was not on the analyte list during wastewater testing.

Water releases:

No data available.

Cottage Grove, MN

Engineering calculations are used to estimate the amount of material discharged to wastewater and/or waste.

Estimated Total
Annual Releases

Estimated %
Accuracy of

(lbs.) (1997)

Estimate (optional)

Water releases:

No data available.

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

C. **ON-SITE LAND RELEASES**

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation.

0

	Estimated Total Annual Releases (lbs.) (1997)		Estimated % Accuracy of Estimate (optional)	
Landfill		0		
Land Treatment/Land Amendmen	ıt	No data ava	ilable	
Surface Impoundments		No data ava	ilable/No longer used	

Comments:

Other (specify):

Cottage Grove, MN

Underground Injection

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)
Landfill	0	
Land Treatment/Land Amendment	0	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

D. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfer cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included. Incineration is now the primary disposal method for these wastes.

Decatur, AL

11,000 lbs. of MeFOSA were identified in the waste, but not by specific waste disposal location. A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that 70 is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the predominant disposal method for these materials.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS data available.	
Wastewater Treatment (Excluding POTW)	See above.	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	No specific CAS dat available.	11,000 lbs. of MeFOSA
Other Landfill	No specific CAS data available.	
Recycle or Recovery	No specific CAS data available.	

Unknown or Other

Cottage Grove, MN

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS nu	mber data available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	

Unknown or Other

Comments:

Cottage Grove facility utilizes incineration for all their drummed wastes.

Sludge from the Cottage Grove facility is sent to an industrial landfill.

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

No information available at this time.

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<0.25				
0.25-1	4	4	24	
1-8		24		
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state; vapors are produced from molten material; material concentration ranges from <1% to 80%.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; materials typically present in waste stream at less than 1%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

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There has been area/source air monitoring data and/or surface wipe sampling data collected for this material at the plant. Area/source sample results and/or surface wipe sample results are used to identify areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. Prior to 1999, these samples were considered to be semi-validated.

Surfaces in production and administration areas were sampled in 1998. A glass fiber filter wetted with methyl alcohol was wiped inside a 10 cm x 10 cm square with analysis by LC-MS. Results indicated the presence of these materials on floors and equipment surfaces in production areas. This resulted in improvements to Hazard Communication practices, personal hygiene emphasis, personal protective equipment emphasis, and several engineering and administrative changes.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds, including compounds covered in this submittal. The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

Air Sampling Data for 31506-32-8

Job Classification	Sample number	Concentration (mg/m3)	Minimum (mg/m3)	Maximum (mg/m3)	Geometric Mean	Geometric Standard Deviation
		Totals →	0.005	1.3	0.041	7.1
Chemical Operator Building 3	95-006*	0.009				
Ū	95-007*	0.005				
	95-014*	0.007				
	95-040*	0.42				
	95-041*	0.043				
Chemical Operator Flaker	95-024*	0.011		- 120 - 1111 - 120		
	97-091*	0.03				
Team Leader Building 3	98-481*	1.3				
Team Leader Flaker	98-098*	0.14				

^{*}The value for this sample is one of several fluorochemical analytes collected on the sample.

Samples are all personal breathing zone samples taken over the duration of a specific task.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials. Operator control rooms are segregated from process areas and provided separate room air ventilation.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if d desired.)

Current exposures are expected to be less than those measured in the recent past. Significant sources have been identified and eliminated. In 1998 an extensive area air sampling of the production areas was performed. Areas with higher levels of airborne materials were investigated further to identify sources. These sources, which include the flaking equipment, are in the process of being eliminated or reduced through replacement with better ventilated equipment. In addition, the ventilation system in the primary production building (3) is being upgraded.

The fluorochemical hazard communication program was improved upon in 1998. This has resulted in improved exposure avoidance behaviors among production employees and better use of personal protective equipment requirements.

Additional hand wash facilities have been installed in primary process buildings. Food consumption and other hand-to-mouth activities are prohibited. There is no smoking in process buildings. Process employees are instructed to remove and leave their work boots and work coveralls at the plant. Work coveralls are professionally laundered.

Non-production equipment surfaces such as floors, doorknobs and stair rails are cleaned frequently.

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Intermediate consumed in the production of a polymeric product which is used as a protective treatment for nonwovens.

Product chemical class or product chemical (Include CAS number if appropriate)

1. Fluorochemical Urethane

% of total volume of subject chemical manufactured or imported

82-87%

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate)

1. Not applicable.

% of total
volume of subject
chemical manufactured
or imported

As reported in Part III, p.2

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

Use Number 1 of 1

>90%

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be used as a polymer melt additive to be used as protective, repellent treatments for nonwoven substrates. These substrates will be made into industrial workwear or medical garments to be used by commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: 13-18 ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate Weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)

Voluntary Use and Exposure Information Profile N-Ethylperfluorooctylsulfonamido ethanol

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

N-Ethylperfluorooctylsulfonamido ethanol (EtFOSE)

CAS Number:

1691-99-2

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
1691-99-2*	> 1MM & < 10MM lbs.	0

Estimate the amount of subject chemical distributed off-site:

<.5% of manufacture/import

*Submitted in 1998 TSCA IUR

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluoroctanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

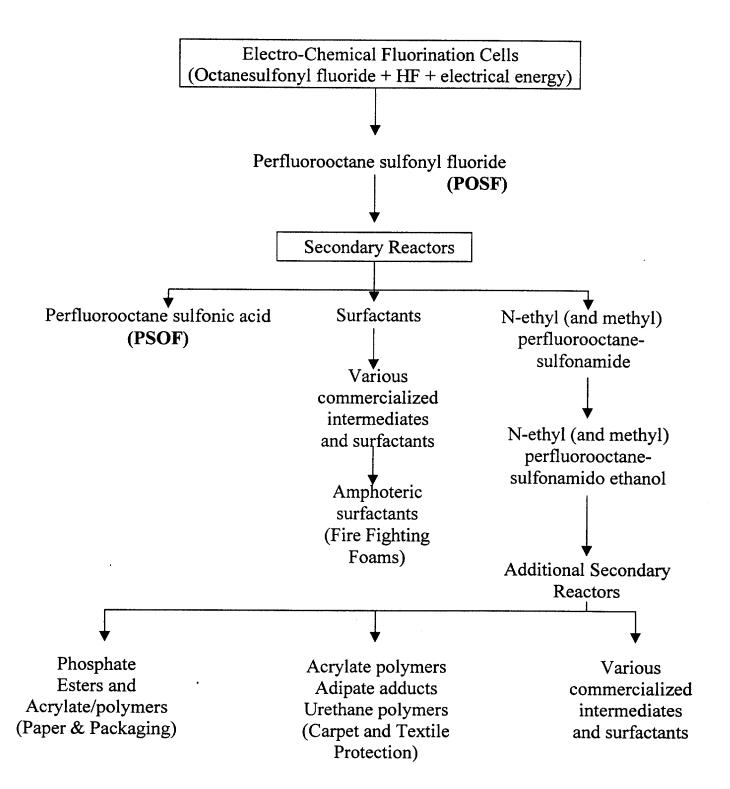
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol.. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS - Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds and is detailed later in this document. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

Decatur, AL

Wastewater fugitive emission data was based upon wastewater testing.

Fugitive emissions may have occurred during some handling steps but have not been quantified.

•	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – wastewater			
	< 1 in 1997	7	250
	0 in 1999)	

Decatur, AL

Stack (point)

Engineering calculations and models of process vent emissions are used for estimates. Existing process emission information shows only minor losses through point source vents.

Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
<1		250

Cottage Grove

Emissions estimates are from process engineer's estimates and emission models.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive (non-point)	No data available.		< 10
Stack (point)			
EtFOSE	< 1		< 10

B. WATER RELEASES FROM SITE

Decatur, AL

The data presented is part of wastewater testing conducted in 1998, since the individual compounds of interest were not analyzed in the 1997 wastewater material balance due to method availability and initial program focus. The 1998 data has not been adjusted for production levels in 1997, since most production remained at similar levels.

It is not possible to distinguish the amount of chemicals generated from specific reaction steps in production of various compounds, or whether their presence may be the result of hydrolysis of more complex molecules.

Estima	ated Total	Estimated %
Annua	l Releases	Accuracy of
(lbs.)	(1997)	Estimate (optional)

Water releases:

45

Number of days/year release occurs: Most compounds are manufactured on a regular basis, so releases are estimated at 250 days per year.

Receiving Water Name: Baker's Creek at the junction with the Tennessee River

NPDES Number: ALD004023164

Comments:

Cottage Grove, MN

Engineering calculations were used to estimate the amount of material discharged to wastewater. The amount of material discharged to the river was determined through use of existing removal efficiency testing results from another facility.

Estimated Total	Estimated %
Annual Releases	Accuracy of
(lbs.) (1997)	Estimate (optional)

Water releases:

0

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation.

		ated Total al Releases	Estimated % Accuracy of
	(lbs.)	(1997)	Estimate (optional)
Landfill		0	
Land Treatment/Land Amendmen	t	15800 – No	longer used
Surface Impoundments		No data avai	lable/No longer used
Underground Injection		0	_
Other (specify):			

Comments:

Cottage Grove, MN

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

	Estimated Total Annual Releases	Estimated % Accuracy of
	(lbs.) (1997)	Estimate (optional)
Landfill	0	
Land Treatment/Land Amendment	0	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

C. **OFF-SITE TRANSFERS**

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name: Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information:

There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

45,700 lbs. of EtFOSE were identified in the waste, but not by specific waste disposal location. A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the predominant disposal method for these materials.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS data available.	
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	No specific CAS data available.	45,700 lbs. of EtFOSE
Other Landfill	No specific CAS data available.	
· Recycle or Recovery	0	
Unknown or Other	0	/

Cottage Grove, MN

Comments:

Cottage Grove facility utilizes incineration for all their drummed wastes.

,	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS da	nta available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other	0	

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<.25	14			
.25-1	12			
1-8	12			
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state or dissolved in organic solvents; vapors are produced from molten material; approximate concentration ranges from <1% to 80%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Positionable local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Process operating standards list the respirator (e.g., supplied air, organic vapor cartridge with particulate prefilter, or particulate filtering), glove by elastomer (e.g., neoprene or nitrile), chemical protective clothing (e.g., 2-piece PVC rainsuit), eye protection (e.g., chemical splash goggles with or without full faceshield depending on type of respirator used) to be used by the employee when the task involves exposure to a particular fluorochemical material.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<0.25				
0.25-1	4	4	24	
1-8		48	24	
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, flaking monomer, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state; vapors are produced from molten material; material concentration ranges from <1% to 80%. Exposure also occurs when acrylate fluorochemical monomers containing <2% 1691-99-2 are flaked.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; materials typically present in waste stream at less than 1%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

Personal sampling for these materials is currently (late 1999 to present) done using OSHA Versatile Sampler tubes with XAD-4 resin and mixed cellulose ester or glass fiber prefilter. Sample analysis is by LC-MS. Previous sampling had employed silica gel acid mist tubes with GC-ECD analysis. See attached table for air sample results.

There has been area/source air monitoring data and/or surface wipe sampling data collected for this material at the plant. Area/source sample results and/or surface wipe sample results are used to identify areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. Prior to 1999, these samples were considered to be semi-validated.

Surfaces in production and administration areas were sampled in 1998. A glass fiber filter wetted with methyl alcohol was wiped inside a 10 cm x 10 cm square with analysis by LC-MS. Results indicated the presence of these materials on floors and equipment surfaces in production areas. This resulted in improvements to Hazard Communication practices, personal hygiene emphasis, personal protective equipment emphasis, and several engineering and administrative changes.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds, including compounds covered in this submittal. The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

Air Sampling Data for 1691-99-2

Job Classification	Sample number	Concentration (mg/m3)	Minimum (mg/m3)	Maximum (mg/m3)	Geometric Mean	Geometric Standard Deviation
.		Totals	<0.005	0.08	0.01	2.7
Chemical Operator						
Building 3	94-001	0.08				
Daliding 5	94-001	0.019				
	94-003	0.047				
	94-031*	<0.01				
	94-032*	<0.01				
	94-034*	<0.005				
	94-037*	0.008				
	94-038*	0.003				
	94-039*	<0.007				
	94-041*	<0.01				
	94-046*	<0.005				
	94-050*	< 0.005				
	95-041*	0.07				
Team Leader Building 3	94-036*	0.003				
J	94-047*	<0.02				
	94-051*	<0.005				
	94-054*	0.006				
	94-055*	<0.005				
	J-7-000	٠٥.005				

^{*} The value for this sample is one of several fluorochemical analytes collected on the sample.

Samples are all personal breathing zone samples taken over the duration of a specific task.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. Some equipment, such as the flaker, is equipped with local exhaust ventilation. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Current exposures are expected to be less than those measured in the recent past. Significant sources of exposure have been identified and eliminated. In 1998 an extensive area air sampling of the production areas was performed. Areas with higher levels of airborne materials were investigated further to identify sources. These sources, which include the flaking equipment, are in the process of being eliminated or reduced through replacement with better ventilated equipment. In addition, the ventilation system in the primary production building (3) is being upgraded.

The fluorochemical hazard communication program was improved upon in 1998. This has resulted in improved exposure avoidance behaviors among production employees and better use of personal protective equipment requirements.

Additional hand wash facilities have been installed in primary process buildings. Food consumption and other hand-to-mouth activities are prohibited. There is no smoking in process buildings. Process employees are instructed to remove and leave their work boots and work coveralls at the plant. Work coveralls are professionally laundered.

Non-production equipment surfaces such as floors, doorknobs and stair rails are cleaned frequently.

VII. **CHEMICAL END USES**

END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS A.

A1. On-Site Use as a Intermediate:

Intermediate consumed in the production of several polymeric products which are used a protective treatments for upholstery, apparel, leather, carpeting, and paper packaging. Also used as an intermediate to manufacture materials used in floor polish and coating additives.

(Incl	Product chemical class or product chemical ude CAS number if appropriate)	% of total volume of subject chemical manufactured or imported
1.	Fluorochemical Urethanes	5-10%
2.	Fluorochemical Esters	< 1%
3.	Fluorochemical Allophanate	5-10%
4.	Fluorochemical Salt CAS #30381-98-7	75-80%
5.	Fluorinated Alkylpolyoxyethylene Ethanols	1-3%
		As reported in Part III, p.2
	Product chemical class or product chemical	% of total volume of subject chemical manufactured

(Include CAS number if appropriate)

or imported

1.	N-ethyl perfluorooctanesulfonamidoethyl acrylate
	CAS #423-82-5

< 2-6%

2. N-ethyl perfluorooctanesulfonamidoethyl methacrylate CAS #376-14-7

< 1%

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate)

% of total volume of subject chemical manufactured or imported

1. Not applicable.

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

Use Number 1 of 7

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be mill applied as protective, repellent treatments for apparel substrates. These substrates will be made into apparel products to be used by consumers and commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:		
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vaporX Liquid solution Other (Explain)		
Use Number 2 of 7 Description of Chemical End Use: This CAS numanufacture polymers that are sold to industrial us repellent treatments for apparel substrates. These products to be used by consumers and commercial present in these products at low levels as a residual	ers to be mill applied as protective, substrates will be made into apparel industry. This CAS number itself will be		
Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:		
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: X <1% (< 1000 ppm) 1-30% 30-60% 60-90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)		
>90%			

Use Number 3 of 5

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be mill applied as protective, repellent treatments for leather substrates. These substrates will be made into apparel products to be used by consumers and commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: <1 \pm	Check all physical forms of the chemical during this use:	
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: X <1% (< 1300 ppm) 1-30% 30-60% 60-90% >90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)	
Use Number 4 of 5 Description of Chemical End Use: This CAS number annufacture chemical substances that are regulated indirect food additives used to impart grease and oil Paper or paperboard are used in food packaging protrays. This CAS number itself will be present in the	by the Food and Drug Administration as resistance to paper and paperboard. ducts such as bags, wrappers, cartons, or	
Percent of total manufactured or imported volume going to this use: <1 \pm	Check all physical forms of the chemical during this use:	
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid	
X <1% (< 2000 ppm) 1-30% 30-60% 60-90% >90%	Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)	

Use Number 5 of 7

Description of Chemical End Use:	This CAS number is used as an intermediate to
manufacture chemical substances tha	at are used in floor polishes.

Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:	
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: <1% 1-30% (4%) 30-60% 60-90% >90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vaporX Liquid solution Other (Explain)	
Use Number 6 of 7 Description of Chemical End Use: This CAS number annufacture chemical substances that are used in c		
Percent of total manufactured or imported volume going to this use: <1 \pm	Check all physical forms of the chemical during this use:	
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet soli Gas or vapor X Liquid solution Other (Explain)	

Use Number 7 of 7

Description of Chemical End Use: This CAS number is used as an intermediate in the industrial synthesis of chemicals and fluoropolymers used as textile treating resins, paper chemicals, and/or surfactants.

Percent of total manufactured or imported volume going to this use: <1 \pm	Check all physical forms of the chemical during this use:	
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vaporX Liquid solution Other (Explain)	

(a) invitation

Chairperson:

Karin L. Beaver

Start:

06/01/2000 01:00 PM

End:

06/01/2000 01:30 PM

Description:

Bill Weppner & Paul Heerwald to discuss Novapharm

invitees:

Paul E. Heerwald/US-Corporate/3M/US Bill Weppner/US-Corporate/3M/US

Detailed description:

1

Voluntary Use and Exposure Information Profile N-methyl perfluorooctanesulfonamido ethanol

I. CHEMICAL IDENTIFICATION

Chemical Name:

N-methyl perfluorooctanesulfonamido ethanol (MeFOSE)

CAS Number:

24448-09-7

П. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

22614 Route 84 North

Cordova, IL 61242

10746 Innovation Road Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
24448-09-7*	> 1MM & < 10MM lb.	0

Estimate the amount of subject chemical distributed off-site: <2%

^{*}Submitted in 1998 TSCA IUR.

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluorocatanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

$$\begin{array}{c} Energy \\ C_8H_{17}S0_2F \ + \ 17\ HF \\ ECF \\ \hline \\ 1\text{-Octanesulfonyl fluoride} \\ \end{array}$$

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

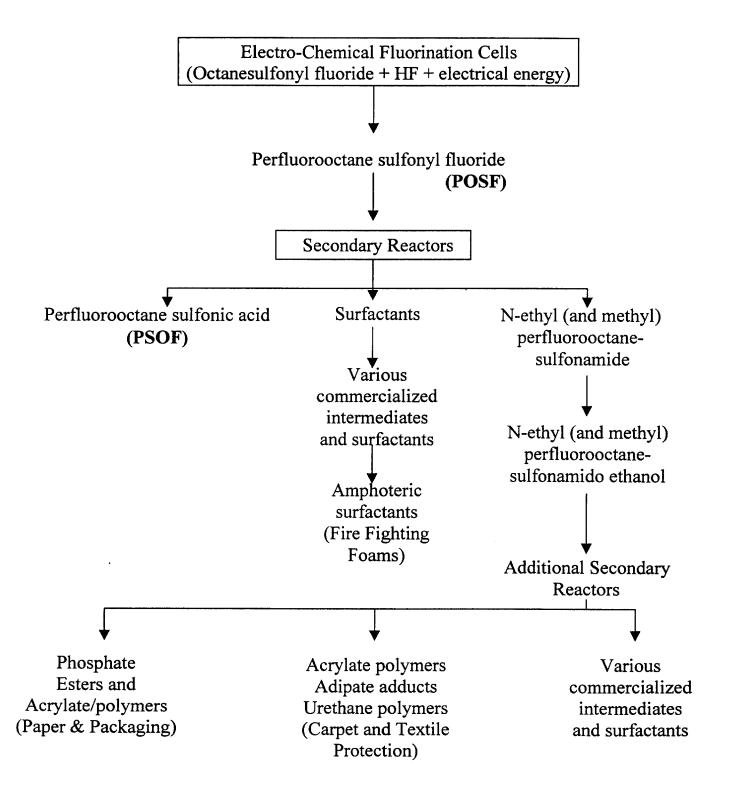
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS - Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

Decatur, AL

Wastewater fugitive emission data was based upon wastewater testing.

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

•			
	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – wastewater	< 1		
Stack (point)			
	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
	< 1		250

Cottage Grove

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive (non-point)	0		
Stack (point)	0	,	

Cordova, IL

POSF-based products are no longer manufactured in Cordova. Fugitive emissions may have occurred during some handling steps but have not been quantified.

	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive (non-point)	No data available.		

No data available.

Comments:

Stack (point)

B. WATER RELEASES FROM SITE

Decatur, AL

The data presented is part of wastewater conducted in 1998, since the individual compounds of interest were not analyzed in the 1997 wastewater material balance due to method availability and initial program focus. The 1998 data has not been adjusted for production levels in 1997, since most production remained at similar levels.

It is not possible to distinguish the amount of chemicals generated from specific reaction steps in production of various compounds, or whether their presence may be the result of hydrolysis of more complex molecules.

Estimated Total Estimated %
Annual Releases (lbs.) (1997) Estimate (optional)

Water releases:

170

Number of days/year release occurs: 250

Receiving Water Name: Baker's Creek at the junction with the Tennessee River

NPDES Number: ALD004023164

Comments:

Cordova, IL

Engineering calculations are used to estimate the amount of material discharged to wastewater.

Estimated Total Estimated %
Annual Releases (lbs.) (1997) Estimate (optional)

Water releases:

< 10

Number of days/year release occurs: 1 time in 1997. POSF-based products no longer produced in Cordova.

Receiving Water Name: Mississippi River

NPDES Number: IL0003140

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation. Levels of the compound in the sludge were determined from wastewater data.

Estimated Total	Estimated %
Annual Releases	Accuracy of
(lbs.) (1997)	Estimate (optional)

Landfill 0

Land Treatment/Land Amendment 25,500 – No longer used **Surface Impoundments** No data available/No longer used

Underground Injection 0
Other (specify): 0

Comments:

Cordova, IL

Less than 10 pounds total of all compounds per year are released to the Cordova wastewater system.

The sludge formed in the primary clarifier is most likely to contain the insoluble sulfonated materials. This sludge is removed and incinerated. The sludge impoundments are lined.

The manufacture of MeFOSE has been discontinued at so these releases have also been discontinued.

	Estimated Total Annual Releases	Estimated % Accuracy of Estimate (optional)
	(lbs.) (1997)	
Landfill	0	
Land Treatment/Land Amendmen	nt < 10	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

Cottage Grove, MN

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill.

	Estimated Total Annual Releases	Estimated % Accuracy of Estimate (optional)
	(lbs.) (1997)	
Landfill	0	
Land Treatment/Land Amendment	0	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

Comments:

D. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name: Street Address:

City: Country: State: Zip Code:

NPDES Number:

Cordova, IL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name: Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

35,000 lbs. of MeFOSE identified in the waste, but not by specific waste disposal location. A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the primary disposal method for these wastes.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS data available.	
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	No specific CAS data available.	Total 35,000 lbs. of MeFOSE
Other Landfill	No specific CAS data available.	
Recycle or Recovery	0	
Unknown or Other	0	
Comments:		

Cordova, IL

Cordova wastes that contained MeFOSE were all incinerated. Wastes included some off spec product, empty bags and empty drums and contained the compounds is residual amounts. These products are no longer made at Cordova.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS dat	a available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	· O	
Recycle or Recovery	0	
Unknown or Other	0	
Comments:		

Cottage Grove, MN

Cottage Grove facility utilizes incineration for all their drummed wastes.

. Sludge from the Cottage Grove facility is sent to an industrial landfill.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	60	
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
	146	

CAS Number 24448-09-7 3M Company
Other Landfill 0

Recycle or Recovery 0

Comments:

VI. ON-SITE WORKPLACE EXPOSURE

Unknown or Other

3M Company, Specialty Materials Manufacturing Division, Cordova, IL

POSF-based products are no longer manufactured at Cordova.

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

0

13

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<.25	12			
.25-1	12			
1-8	12			
. >8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state or dissolved in organic solvents; vapors are produced from molten material; approximate concentration ranges from <1% to 80%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Positionable local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Process operating standards list the respirator (e.g., supplied air, organic vapor cartridge with particulate prefilter, or particulate filtering), glove by elastomer (e.g., neoprene or nitrile), chemical protective clothing (e.g., 2-piece PVC rainsuit), eye protection (e.g., chemical splash goggles with or without full faceshield depending on type of respirator used) to be used by the employee when the task involves exposure to a particular fluorochemical material.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year			
Hours/Day	<10	10-100	100-250	>250
<0.25				
0.25-1	4	4	24	
1-8		48	24	
>8				

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, flaking monomer, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state; vapors are produced from molten material. Material concentration ranges from <1% to 80%. Exposure also occurs when acrylate fluorochemical monomers containing <2% 24448-09-7 are flaked.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; materials typically present in waste stream at less than 1%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

Personal sampling for these materials is currently (late 1999 to present) done using OSHA Versatile Sampler tubes with XAD-4 resin and mixed cellulose ester or glass fiber prefilter. Sample analysis is by LC-MS. Previous sampling had employed silica gel acid mist tubes with GC-ECD analysis. See attached table for air sample results.

There has been area/source air monitoring data and/or surface wipe sampling data collected for this material at the plant. Area/source sample results and/or surface wipe sample results are used to identify areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. Prior to 1999, these samples were considered to be semi-validated.

Surfaces in production and administration areas were sampled in 1998. A glass fiber filter wetted with methyl alcohol was wiped inside a 10 cm x 10 cm square with analysis by LC-MS. Results indicated the presence of these materials on floors and equipment surfaces in production areas. This resulted in improvements in Hazard Communication practices, personal hygiene emphasis, personal protective equipment emphasis, and several engineering and administrative changes.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds, including compounds covered in this submittal. The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

Air Sampling Data for 24448-09-7

Job Classification	Sample number	Concentration (mg/m3)	Minimum (mg/m3)	Maximum (mg/m3)	Geometric Mean	Geometric Standard Deviation
		Totals →	<0.002	1.5	0.013	7.1
Chemical Operator Building 3	94-031*	<0.01				
J	94-032*	<0.01				
	94-034*	<0.005				
	94-037*	0.004				
	94-038*	0.003				
	94-039*	<0.006				
	94-041*	<0.01				
	94-046*	<0.005				
	94-050*	0.01				
	95-006*	< 0.003				
	95-007*	< 0.003				
	95-014*	<0.002				
	95-040*	0.37				
Chemical	95-024	<0.003		- hid is will		
Operator Flaker						
	97-091	0.15				
Team Leader Building 3	94-036	<0.003				
	94-047*	<0.02				
	94-051*	<0.005				
	94-054*	<0.005				
	94-055*	0.005				
	98-481	1.5				
Team Leader Flaker	98-098	0.031				

^{*} The value for this sample is one of several fluorochemical analytes collected on the sample. Samples are all personal breathing zone samples taken over the duration of a specific task.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. Some equipment, such as the flaker, is equipped with local exhaust ventilation. General room air provides for dilution of airborne materials. Operator control rooms are segregated from process areas and provided separate room air ventilation.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

Current exposures are expected to be less than those measured in the recent past. Significant sources of exposure have been identified and eliminated. In 1998 an extensive area air sampling of the production areas was performed. Areas with higher levels of airborne materials were investigated further to identify sources. These sources, which include the flaking equipment, are in the process of being eliminated or reduced through replacement with better ventilated equipment. In addition, the ventilation system in the primary production building (3) is being upgraded.

The fluorochemical hazard communication program was improved upon in 1998. This has resulted in improved exposure avoidance behaviors among production employees and better use of personal protective equipment requirements.

Additional hand wash facilities have been installed in primary process buildings. Food consumption and other hand-to-mouth activities are prohibited. There is no smoking in process buildings. Process employees are instructed to remove and leave their work boots and work coveralls at the plant. Work coveralls are professionally laundered.

Non-production equipment surfaces such as floors, doorknobs and stair rails are cleaned frequently.

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Intermediate consumed in the production of several polymeric products which are used as protective treatments for carpeting, upholstery, and leather.

(Incl	Product chemical class or product chemical lude CAS number if appropriate)	volume of subject chemical manufactured or imported		
1.	Fluorochemical Adipate	35-40%		
2.	Fluorochemical Urethane	1-5%		
3.	Fluorochemical Ester	1-5%		
4.	Fluorochemical Ester	< 1%		

Intermediate consumed in the production of other intermediates to be further reacted to form polymeric materials.

Product chemical class
or product chemical
(Include CAS number if appropriate)

% of total volume of subject chemical manufactured or imported

50-55%

% of total

CAS 25268-77-3		

N-methyl perfluorooctanesulfonamidoethyl acrylate

2. N-methyl perfluorooctanesulfonamidoethyl methacrylate 1-5% CAS 14650-24-9

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total
volume of subject
chemical manufactured
or imported

1. Not applicable.

1.

As reported in Part III, p.2

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

Use Number 1 of 5

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be mill applied as protective, repellent treatments for upholstery. The upholstery will be used by consumers and commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: 1-2	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: \[\frac{X}{2} < 1\% \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)
Use Number 2 of 3 Description of Chemical End Use: This CAS number annufacture polymers that are sold to industrial use repellent treatments for carpeting. The upholstery vindustry. This CAS number itself will be present in Percent of total manufactured or imported volume going to this use: <2 ±	ers to be mill applied as protective, will be used by consumers and commercial
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)

Use Number 3 of 5

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be mill applied as protective, repellent treatments for upholstery. The upholstery will be used by consumers and commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: $< 0.1 \pm$	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: \[\frac{X}{2000 \text{ ppm}} \] \[\frac{1\%}{30-60\%} \] \[\frac{60-90\%}{2000} \] \[\frac{90\%}{30-60\%} \]	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vaporX Liquid solution Other (Explain)
Use Number 4 of 5 Description of Chemical End Use: This CAS number manufacture polymers that are sold to industrial use repellent treatments for upholstery. The upholstery commercial industry. This CAS number itself will as a residual.	ers to be mill applied as protective, will be used by consumers and
Percent of total manufactured or imported volume going to this use: $< 0.1 \pm$	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: \[\frac{X}{2} < 1\% \\ \frac{1-30\%}{30-60\%} \\ \frac{60-90\%}{20-90\%} \] \[\frac{30-60\%}{20-90\%} \]	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)

Use Number 5 of 5

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be mill applied as protective, repellent treatments for upholstery. The upholstery will be used by consumers and commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: <2 ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid
<1%	Gas or vapor
1-30%	X_ Liquid solution
30-60%	Other (Explain)
1-30% 30-60% X 60-90%	
>90%	

Voluntary Use and Exposure Information Profile N-ethylperfluorooctylsulfonamido ethyl acrylate

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

N-ethylperfluorooctylsulfonamido ethyl acrylate (EtFOSEA)

CAS Number:

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
423-82-5	< 100M lbs.	0

Estimate the amount of subject chemical distributed off-site: 100%

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluorocatanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

Energy
$$C_8H_{17}S0_2F + 17 HF$$
 $C_8F_{17}S0_2F + 17 H_2$
ECF

1-Octanesulfonyl fluoride

Perfluorooctanesulfonyl fluoride

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

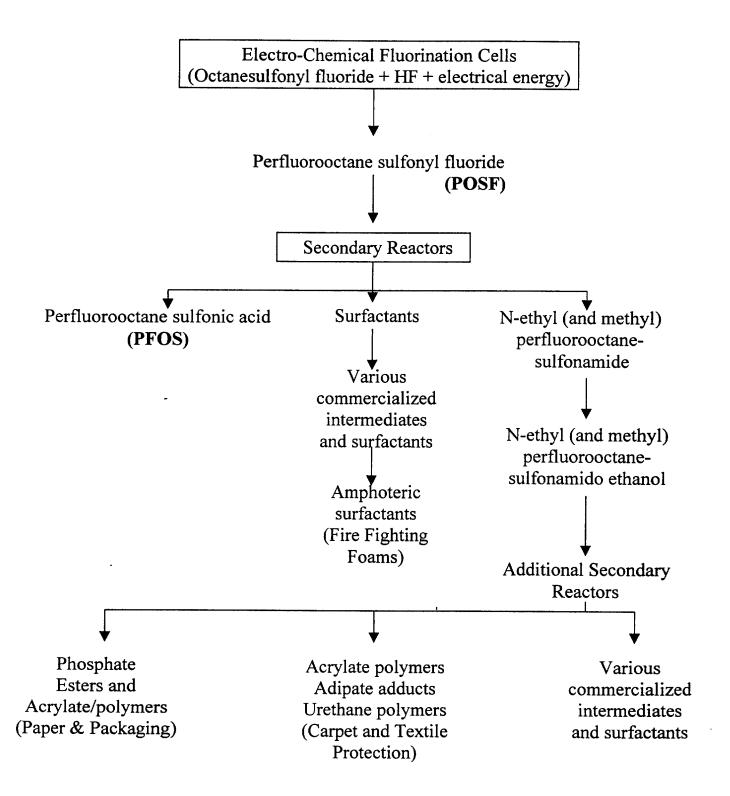
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

A. ON-SITE AIR RELEASES

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

ALL PLANTS – Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

Decatur, AL

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – (non-point)	No data available.		
Stack (point)			
	Estimated Total Annual Releases	Estimated % Accuracy of	# days/years release occurs

(lbs.) (1997)

Estimate (optional)

CAS Number 423-82-5

3M Company

6

Cottage Grove

Emissions estimates are from process engineer's estimates and emission models.

Estimated Total Annual Releases Estimated %
Accuracy of
Estimate (optional)

days/years release occurs

(lbs.) (1997)

Fugitive (non-point)

No data available.

Stack (point)

0

В. WATER RELEASES FROM SITE

Decatur, AL

The data presented is part of wastewater testing conducted in 1998, since the individual compounds of interest were not analyzed in the 1997 wastewater material balance due to method availability and initial program focus. The 1998 data has not been adjusted for production levels in 1997, since most production remained at similar levels.

Materials in the sludge and wastewater may result from production losses in the manufacturing of these compounds or from the hydrolysis of more complex molecules.

> **Estimated Total Annual Releases** (lbs.) (1997)

Estimated % Accuracy of Estimate (optional)

Water releases:

< 20

Number of days/year release occurs:

2/year

Receiving Water Name:

Baker's Creek at the junction with the Tennessee River

NPDES Number: ALD 004023164

Comments:

Cottage Grove, MN

1800 pounds per year of N-ETFOSEA were estimated discharged to the wastewater system in 1998. No data is available for 1997. Based upon insolubility and removal of similar compounds in the Decatur wastewater treatment facility, the level of this compound in the river discharge is believed to be close to zero (<1% of total discharged).

· Continued improvements in reducing material to the river are being undertaken by a wastewater emission reduction team or engineers at the facility.

> **Estimated Total** Annual Releases (lbs.) (1997)

Estimated % Accuracy of Estimate (optional)

Water releases:

<18

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

C. **ON-SITE LAND RELEASES**

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation.

	Annu	ated Total al Releases (1997)	Estimated % Accuracy of Estimate (optional)
Landfill		0	
Land Treatment/Land Amendmen	it	No data ava	ilable/No longer used
Surface Impoundments		No data ava	ilable/No longer used
Underground Injection		0	

Other (specify):

Comments:

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

Cottage Grove, MN

	Estimated Total Annual Releases	Estimated % Accuracy of
	(lbs.) (1997)	Estimate (optional)
Landfill	0	
Land Treatment/Land Amendment	0	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

D. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the primary disposal method for these materials.

Estimated Total Annual Releases (lbs.)

Estimated %
Accuracy of
Estimate (optional)

Incineration:

No specific CAS data available.

Wastewater Treatment (Excluding POTW)

0

Underground Injection

0

Hazardous Waste (RCRA Subtitle C) landfill

No specific CAS data available.

Other Landfill

No specific CAS data available.

Recycle or Recovery

0

Unknown or Other

Cottage Grove, MN

Cottage Grove facility utilizes incineration for all their drummed wastes.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS da	ta available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other		
Comments:		

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year				
Hours/Day	<10	10-100	100-250	>250	
<.25	2				
.25-1	2				
1-8	2				
>8					

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state or dissolved in organic solvents; vapors are produced from molten material; approximate concentration ranges from <1% to 85%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Positionable local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Process operating standards list the respirator (e.g., supplied air, organic vapor cartridge with particulate prefilter, or particulate filtering), glove by elastomer (e.g., neoprene or nitrile), chemical protective clothing (e.g., 2-piece PVC rainsuit), eye protection (e.g., chemical splash goggles with or without full faceshield depending on type of respirator used) to be used by the employee when the task involves exposure to a particular fluorochemical material.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

Hours/Day	Days/Year				
	<10	10-100	100-250	>250	
<0.25					
0.25-1	4	4			
1-8		48	24		
>8					

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, flaking monomer, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state; vapors are produced from molten material; material concentration ranges up to 85%.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; material typically present in waste stream at less than 1%.

f the

16

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

Personal sampling for these materials is currently (late 1999 to present) done using OSHA Versatile Sampler tubes with XAD-4 resin and mixed cellulose ester or glass fiber prefilter. Sample analysis is by LC-MS. Previous sampling had employed silica gel acid mist tubes with GC-ECD analysis. See attached table for air sample results.

There has been area/source air monitoring data and/or surface wipe sampling data collected for this material at the plant. Area/source sample results and/or surface wipe sample results are used to identify areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. Prior to 1999, these samples were considered to be semi-validated.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds, including compounds covered in this submittal. The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

Air Sampling Data for 423-82-5

Job Classification	Sample number	Concentration (mg/m3)	Minimum (mg/m3)	Maximum (mg/m3)	Geometric Mean	Geometric Standard Deviation
Chemical Operator Building 3	94-031*	Totals → <0.01	<0.002	1.5	0.013	7.1
Danding 0	94-032*	<0.01				
	94-034*	<0.005				
	94-037*	0.004				
	94-038*	0.003				
	94-039*	<0.006				
	94-041*	<0.01				
	94-046*	<0.005				
	94-050*	0.01				
	95-006*	< 0.003				
	95-007*	< 0.003				
	95-014*	<0.002				
	95-040*	0.37				
Chemical	95-024	<0.003				
Operator Flaker						
	97-091	0.15				
Team Leader Building 3	94-036	<0.003				
	94-047*	<0.02				
	94-051*	<0.005				
	94-054*	< 0.005				
	94-055*	0.005				
	98-481	1.5				
Team Leader Flaker	98-098	0.031				

^{*}The value for this sample is one of several fluorochemical analytes collected on the sample. Samples are all personal breathing zone samples taken over the duration of a specific task.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. Some equipment, such as the flaker, is equipped with local exhaust ventilation. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Not applicable.

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Not applicable.

As reported in Part III, p.2

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

Use Number 1 of 1

Description of Chemical End Use: Customers use as an intermediate to make fluoropolymers.

Percent of total manufactured or imported volume going to this use: 100%

Check all physical forms of the chemical during this use:

If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:

 Aerosoi			
 Dry Powd			

<u>X</u> <1% ___ 1-30% ___ Dry Powder
___ Pellets or large crystals

___ 30-60% ___ 60-90% Water or solvent – wet solid
Gas or vapor

___ 60-90% __ >90% <u>Liquid solution</u>Other (Explain)

Voluntary Use and Exposure Information Profile N-ethyl perfluorooctanesulfonamido ethyl methacrylate

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

N-ethyl perfluorooctanesulfonamido ethyl methacrylate (EtFOSEMA)

CAS Number:

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	<u>Imported</u>
376-14-7*	< 100M lbs.	0

Estimate the amount of subject chemical distributed off-site: 0

^{*}Submitted in 1998 TSCA IUR.

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

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Reaction 1

$$\begin{array}{c} Energy \\ C_8H_{17}S0_2F \ + \ 17\ HF \\ \hline ECF \\ 1\text{-Octanesulfonyl fluoride} \\ \end{array}$$

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
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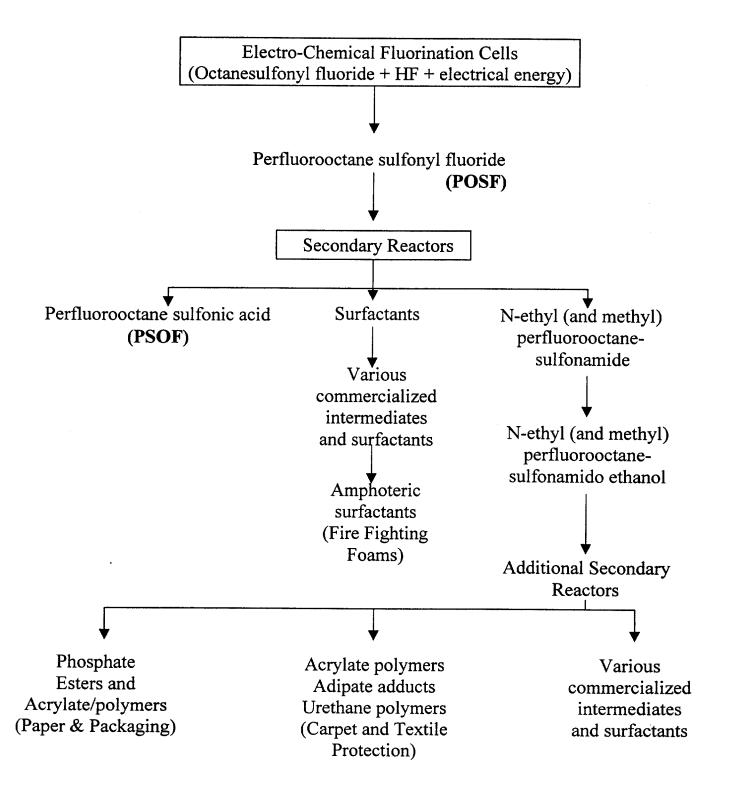
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POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

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Decatur, AL

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – (non-point)	No data available.		
Stack (point)			
	Estimated Total Annual Releases (lbs.) (1997)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs

Cottage Grove

Emission estimates are based upon existing process models and engineering calculations from the facility. Fugitive emissions may have occurred during some handling steps but have not been quantified.

Estimated Total Annual Releases	Estimated % Accuracy of	# days/years release occurs
(lbs.) (1997)	Estimate (optional)	release occurs

Fugitive (non-point)

No data available.

Stack (point)

0

B. WATER RELEASES FROM SITE

Decatur, AL

This chemical was not on the analyte list for wastewater treatment.

Estimated Total Annual Releases Estimated %
Accuracy of

(lbs.) (1997)

Estimate (optional)

Water releases:

No data available.

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

Comments:

Cottage Grove, MN

Engineering calculations were used to estimate the amount of material discharged to wastewater. The amount of material discharged to the river was determined through use of existing removal efficiency testing results from another facility.

Estimated Total Annual Releases (lbs.) (1997)

Estimated %
Accuracy of
Estimate (optional)

Water releases:

0

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation.

Estimated Total	Estimated %
Annual Releases	Accuracy of
(lbs.) (1997)	Estimate (optional)

Landfill

Land Treatment/Land Amendment Surface Impoundments Underground Injection 0 No data available/No longer used No data available/No longer used

Other (specify):

Comments:

Cottage Grove, MN

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

	Estimated Total Annual Releases	Estimated % Accuracy of
	(lbs.) (1997)	Estimate (optional)
Landfill	0	
Land Treatment/Land Amendment	0	
Surface Impoundments	0	
Underground Injection	0	
Other (specify):		

D. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

Comments:

A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that over 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the predominant disposal method for these materials.

•	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS nur	nbers available.
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	No specific CAS num	nbers available.
Other Landfill	No specific CAS nun	nbers available.
Recycle or Recovery	0	
Unknown or Other	-	
•		

Cottage Grove, MN

Comments:

Cottage Grove facility utilizes incineration for all their drummed wastes.

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	No specific CAS nu	ımbers available
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other		

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year				
Hours/Day	<10	10-100	100-250	>250	
<.25	6				
.25-1	8				
1-8	8				
>8					

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state or dissolved in organic solvents; vapors are produced from molten material; approximate concentration ranges from <1% to 80%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Positionable local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Process operating standards list the respirator (e.g., supplied air, organic vapor cartridge with particulate prefilter, or particulate filtering), glove by elastomer (e.g., neoprene or nitrile), chemical protective clothing (e.g., 2-piece PVC rainsuit), eye protection (e.g., chemical splash goggles with or without full faceshield depending on type of respirator used) to be used by the employee when the task involves exposure to a particular fluorochemical material.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year				
Hours/Day	<10	10-100	100-250	>250	
<0.25					
0.25-1	4	4			
1-8		48	24		
>8					

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, flaking monomer, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state; vapors are produced from molten material; material concentration ranges up to 85%.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; material typically present in waste stream at less than 1%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. Some equipment, such as the flaker, is equipped with local exhaust ventilation. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desire

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

N-ethyl perfluorooctanesulfonamido ethyl methacrylate is used in four different processes to react or copolymerize yielding the following product chemicals. These chemicals are intermediates for final products listed in the end use section.

Product chemical class or product chemical (Include CAS number if appropriate)		volume of subject chemical manufactured or imported
1.	Fluoromethacrylate oxide	10%
2.	Fluoroaliphatic polymericesters	17%
3.	Fluorinated alkyl ester	12%
4.	Fluorinated alkyl acrylate	60%

As reported in Part III, p.2

% of total

A2. Off-Site Use as an Intermediate

Used as a reactive intermediate in the industrial synthesis of chemicals and fluoropolymers used as textile treating resins, paper sizings and surfactants.

Product chemical class
or product chemical
(Include CAS number if appropriate)

% of total volume of subject chemical manufactured or imported

1. Fluorinated alkyl methacrylate

< 1%

As reported in Part III, p.2

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

T	Īce	N	ıım	ber	1	οf	3
Ł	.50	1.4	шп	-	1	O.	

>90%

Description of Chemical End Use: Fire fighting professionals use fire fighting foams to aid wetting of water for fire extinguishment and vapor suppression. This chemical is present as a low level residual.

Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid	
Gas or vapor Liquid solution Other (Explain)	
acturers use this chemical as a surfactant to	0
Check all physical forms of the chemical during this use:	
Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor _X Liquid solution Other (Explain)	
ac	X Liquid solution Other (Explain) cturers use this chemical as a surfactant to the chemical during this use: Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution

Use Number 3 of 3

Description of Chemical End Use: Film manufacturers use this chemical as a surfactant to improve the wetting of protective coatings.

	nt of total manufactured or imported ne going to this use: < 1	Check all physical forms of the chemical during this use:		
to ind	d in a mixture check appropriate box icate weight fraction. Average are acceptable:		Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid	
<u>X</u>	<1%		Gas or vapor	
	1-30%	<u>X</u>	Liquid solution	
	30-60%		Other (Explain)	
	60-90%			
	>90%			

Voluntary Use and Exposure Information Profile N-Methyl perfluorooctanesulfonamido ethyl acrylate

I. **CHEMICAL IDENTIFICATION**

Chemical Name:

N-Methyl perfluorooctanesulfonamidoethyl acrylate (MeFOSEA)

CAS Number:

25268-77-3

II. **COMPANY IDENTIFICATION**

Company Name:

3M

Site Location:

10746 Innovation Road

Cottage Grove, MN 55016

1400 State Docks Road Decatur, AL 35601

Technical Contact: W.A. Weppner

Phone:

651/733-6374

Address:

3M Center, Building 236-1B-10

St. Paul, MN 55144

III. **ON-SITE ACTIVITIES**

CAS#	Mfg. (1997)	Imported
25268-77-3*	< 1MM lbs.	0

Estimate the amount of subject chemical distributed off-site: <1%

^{*}Submitted in 1998 TSCA IUR.

Narrative Description and Process Flow Schematic:

3M Company utilizes a process known as Simons Electro-Chemical Fluorination (ECF) to synthesize organofluorine molecules. In this process, organic feedstocks are dispersed in liquid, anhydrous hydrogen fluoride, and an electric current is passed through the solution, causing the hydrogen atoms on the molecule to be replaced with fluorine. The predominant components of the products created by this process have the same carbon skeletal arrangement as the feedstock used, but with all of the hydrogen atoms replaced by fluorine. However, fragmentation and rearrangement of the carbon skeleton can also occur and significant amounts of cleaved, branched and cyclic structures may be formed. The degree of fluorination of the organic feedstock is also dependent upon the specific carbon chain length of the feedstock and parameters of the ECF process such as electrical current and the length of time the process is run. It is possible to synthesize fully fluorinated or perfluoroorganic molecules where all of the hydrogen atoms of the hydrocarbon feedstock have been replaced by fluorine atoms. Using these perfluoroorganic molecules as basic building blocks, unique chemistries can be created by further reactions with functionalized hydrocarbon molecules.

3M has produced sulfonyl based fluorochemicals commercially for over 40 years using the ECF process. A basic building block of such products and the highest production volume fluorochemical 3M manufactures is perfluoroctanesulfonyl fluoride (POSF). The starting feedstock for this reaction is 1-octanesulfonyl fluoride. (Reaction 1)

Reaction 1

$$\begin{array}{c} \text{Energy} \\ \text{C}_8\text{H}_{17}\text{S}0_2\text{F} + 17\,\text{HF} \\ \text{ECF} \\ \text{1-Octanesulfonyl fluoride} \\ \end{array} \\ \begin{array}{c} \text{Energy} \\ \text{------->} \\ \text{ECF} \\ \text{Perfluorooctanesulfonyl fluoride} \\ \end{array}$$

The electrochemical fluorination process yields about 35%-40% straight chain (normal) POSF, and a mixture of biproducts and waste of unknown and variable composition comprised of the following:

- 1) higher and lower straight-chain homologs, i.e., n-CnF₂n₊₁SO₂F, e.g., C₆F₁₃SO₂F, C₇F₁₅SO₂F, C₉F₁₉SO₂F which comprise about 7% of the process output
- 2) branched-chain, perfluoroalkylsulfonyl fluorides with various chain lengths, about 18-20% of the output
- 3) straight-chain, branched, and cyclic (non-functional) perfluoroalkanes and ethers, which comprise about 20-25% of the output
- 4) "tars" (high molecular weight fluorochemical byproducts) and other byproducts, including molecular hydrogen, which comprise about 10-15% of the output.

Because of slight differences in process conditions, raw materials, and equipment, the mixture produced by the electrochemical fluorination process varies somewhat from lot-to-lot and from plant-to-plant. The product that results from electrochemical fluorination is thus not a pure chemical but rather a mix of isomers and homologues. The commercialized POSF derived products are a mixture of approximately 70% linear POSF derivatives and 30% branched POSF derived impurities.

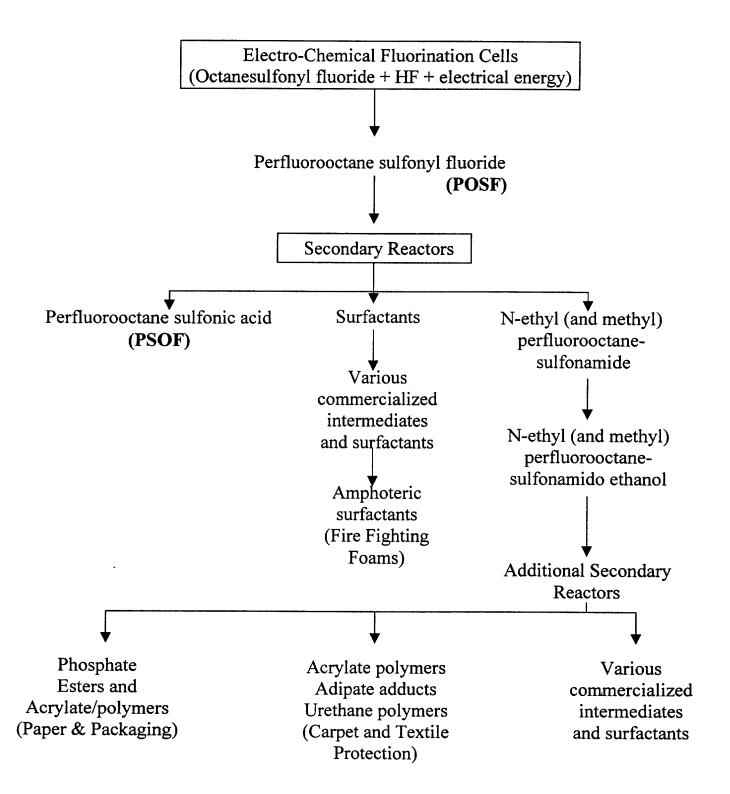
During production, byproducts and waste products are formed. The volatile waste products, such as perfluoromethane, have been vented to the atmosphere in the past, but plans are being implemented to capture and destroy these releases by thermal oxidation over the next few years. The tars have been landfilled in a hazardous waste landfill in the past, but are all currently being incinerated at an in-house or external hazardous waste incinerator. The byproducts, many of which are incompletely fluorinated with hydrogen atoms still present, can be recycled back into the ECF process or are partially degraded in stabilization processes and discharged to controlled, in-house, wastewater treatment systems. The treatment sludge associated with the ECF process has been either landfilled or land-incorporated in the past, but all of this treatment sludge is being landfilled off-site since early 1998.

POSF is itself a commercially viable product, but is primarily an important intermediate in the synthesis of substances used in many other 3M fluorochemical products. The majority is used to produce functionally derivatized fluorochemicals and high molecular weight polymeric products.

Unique chemistries can be created by derivatizing POSF through the sulfonyl fluoride moiety of the molecule using conventional hydrocarbon reactions. POSF is reacted with methyl or ethyl amine to produce either N-methyl or N-ethylperfluorooctanesulfonamide (MeFOSA or EtFOSA). Some of the MeFOSA and EtFOSA is isolated to be used as intermediates to be reacted to form other materials. The majority of the MeFOSA and EtFOSA is not isolated and subsequently reacted with ethylene carbonate to form either N-methyl or N-ethylperfluorooctane sulfonamido ethanol. These are the principal building blocks of the 3M product lines.

The following block flow diagram describes the process discussed above.

BLOCK FLOW DIAGRAM



IV. SITE RELEASE AND TRANSFER INFORMATION FOR TRI CHEMICALS

Not applicable.

V. SITE RELEASE AND TRANSFER INFORMATION FOR NON-TRI CHEMICALS

While analytical methods have improved over time, large variability still exists for certain matrices and compounds, so data available for this report is mostly of a qualitative nature. Although limited monitoring data exists, most emission and waste estimates are based upon process models and engineering calculations. Engineering calculations, however, have limitations with respect to fluorochemicals because fluorochemical losses were not always included in the analysis of each intermediate step.

The accuracy of the emissions data submitted varies due to several factors. Batch process systems are difficult to measure due to quickly changing process conditions, venting pressures and difficulty in isolating processes to take measurements. Additionally, the unique characteristics of these compounds cause them to behave differently from conventional compounds, and physical chemical data properties are not available for all intermediate reaction steps in the past.

A. ON-SITE AIR RELEASES

ALL PLANTS -

Fugitive emissions may occur from vacuum charging from drums, sampling from reactors, drumming of product/intermediate, flaking monomer, drying operations. Materials may be handled in a molten or solid state; vapors are produced from molten material. Industrial hygiene monitoring has been conducted for some compounds. Some minor amounts of these compounds have been detected as fugitive emissions during industrial hygiene exposure testing.

Decatur, AL

Emission estimates are based upon existing process models and engineering calculations.

Fugitive emissions may have occurred during some handling steps but have not been quantified.

·	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)	# days/years release occurs
Fugitive – (non-point)	Not quantified.		
Stack (point)			

Estimated Total Estimated % # days/years
Annual Releases Accuracy of release occurs
(lbs.) (1997) Estimate (optional)

Cottage Grove

Fugitive emissions may have occurred during some handling steps but have not been quantified.

Estimated Total Annual Releases (lbs.) (1997) Estimated % Accuracy of

Estimate (optional)

days/years release occurs

Fugitive (non-point)

No data available.

Stack (point)

No data available.

B. WATER RELEASES FROM SITE

Decatur, AL

This compound was not part of analyte list in wastewater studies.

Estimated Total Annual Releases Estimated % Accuracy of

(lbs.) (1997) Esti

Estimate (optional)

Water releases:

No data available.

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

Comments:

Cottage Grove, MN

Engineering calculations are used to estimate the amount of material discharged to wastewater.

Estimated Total Annual Releases

Estimated % Accuracy of

(lbs.) (1997)

Estimate (optional)

Water releases:

0

Number of days/year release occurs:

Receiving Water Name:

NPDES Number:

C. ON-SITE LAND RELEASES

Decatur, AL

The land treatment of Decatur sludge was discontinued in early 1998. Sludge is now transported to an offsite landfill, after passing through a thickener and a sludge press. An impoundment was used in 1997 as part of the wastewater treatment operation but is now only used for back-up operation.

	Estimated Total Annual Releases		Estimated % Accuracy of	
	(lbs.)	(1997)	Estimate (optional)	
Landfill		0		
Land Treatment/Land Amendmen	t	Not quantific	ed/No longer used	
Surface Impoundments		Not quantific	ed/No longer used	
Underground Injection		0	•	
Other (specify):				

Comments:

Cottage Grove, MN

Sludge from the Cottage Grove facility is sent to an off-site industrial landfill

	Estimated Total Annual Releases		Estimated % Accuracy of
	(lbs.)	(1997)	Estimate (optional)
Landfill		0	
Land Treatment/Land Amendment		0	
Surface Impoundments		0	
Underground Injection		0	
Other (specify):			

. Comments:

D. OFF-SITE TRANSFERS

Decatur, AL

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name:

Street Address:

City:

Country:

State:

Zip Code:

NPDES Number:

Cottage Grove, MN

Process wastewaters are managed in an on-site wastewater treatment facility and are not sent to the POTW.

D1. Transfer to Publicly Owned Treatment Works (POTW)

Number of days/year the release occurs:

Annual Transfer (lb): 0

Estimated % Accuracy of Transfer Estimate (optional) (%):

POTW Name: Street Address:

Sil CCL I

City:

Country:

State:

Zip Code:

NPDES Number:

D2. TRANSFERS TO OTHER OFF-SITE LOCATIONS

General Waste Information: There is limited information by CAS number for compound specific reporting and off-site transfers cannot be readily verified. Rather wastes are classified by halogen content, regulatory waste codes, physical properties and non-specific fluorochemical categories. Where wastes are tracked by CAS number, the amounts have been included.

Decatur, AL

A review of plant records regarding waste disposal locations for Decatur fluoride-containing wastes indicates that 70% is disposed through incineration at various off-site locations and approximately 30% is landfilled at a hazardous waste landfill. Incineration is now the predominant disposal method for these materials.

Estimated Total
Annual Releases
(lbs.)

Estimated %
Accuracy of
Estimate (optional)

Incineration: No specific CAS number data available.

Wastewater Treatment 0 (Excluding POTW)

Underground Injection 0

Hazardous Waste (RCRA No specific CAS number data available. Subtitle C) landfill

Other Landfill No specific CAS number data available.

Recycle or Recovery

Unknown or Other

Cottage Grove facility utilizes incineration for all their drummed wastes.

Cottage Grove, MN

	Estimated Total Annual Releases (lbs.)	Estimated % Accuracy of Estimate (optional)
Incineration:	130	
Wastewater Treatment (Excluding POTW)	0	
Underground Injection	0	
Hazardous Waste (RCRA Subtitle C) landfill	0	
Other Landfill	0	
Recycle or Recovery	0	
Unknown or Other	0	
Comments:		

VI. ON-SITE WORKPLACE EXPOSURE

3M Company, Specialty Materials Manufacturing Division, Cordova, IL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

Hours/Day	Days/Year				
	<10	10-100	100-250	>250	
<.25					
.25-1	4				
1-8					
>8					

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

The chemical (25268-77-3) is an ingredient in the form of a non-dusting, solid, waxy flake (80% concentration) that is charged from fiber drums into a process vessel where it is consumed in a reaction leaving less than 1% residual in the final product.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

No additional engineering controls are used.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

During charging employees wear safety glasses and hard hats, and gloves.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Cottage Grove, MN

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

		Days/Year				
Hours/Day	<10	10-100	100-250	>250		
<.25	4					
.25-1	4					
1-8	4					
>8						

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state or dissolved in organic solvents; vapors are produced from molten material; approximate concentration ranges from <1% to 80%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

There is no chemical-specific personal industrial hygiene monitoring data or area/source monitoring data for this specific material at this facility. For most areas of the facility, recent qualitative assessment of potential exposure to this material under 3M's ongoing industrial hygiene program indicates a low exposure potential to this material.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Positionable local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Process operating standards list the respirator (e.g., supplied air, organic vapor cartridge with particulate prefilter, or particulate filtering), glove by elastomer (e.g., neoprene or nitrile), chemical protective clothing (e.g., 2-piece PVC rainsuit), eye protection (e.g., chemical splash goggles with or without full faceshield depending on type of respirator used) to be used by the employee when the task involves exposure to a particular fluorochemical material.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

3M Company, Specialty Materials Manufacturing Division, Decatur, AL

This information will assist EPA in characterizing the number of workers potentially exposed and the magnitude, frequency, and duration of potential exposure. When providing monitoring data, ensure that data is linked with worker activities described in question 2.

1. Estimate the number of workers potentially exposed routinely to the subject chemical for each of the exposure duration times. If a worker is involved in more than one activity, enter only his/her most typical activity in the table. Don't count a worker more than once. The total number in the table should equal the total number of workers potentially exposed.

	Days/Year				
Hours/Day	<10	10-100	100-250	>250	
<0.25				- 10 th	
0.25-1	4	4			
1-8		48	24		
>8				-	

2. Describe the routine worker activities to which the workers in question 1 are exposed: sampling, removal of filter cake, and drumming of liquids, manufacture an article, etc. For these activities, describe the physical state of the subject chemical (liquid, gas, particulate, or aerosol, etc.) and, if in a mixture, the chemical's concentration:

Vacuum charging from drums, sampling from reactor, drumming of product/intermediate, flaking monomer, maintenance (changing flange, hose, pipe, valve, filter, pump or sight glass); materials may be handled in a molten or solid state; vapors are produced from molten material; material concentration ranges up to 85%.

Wastewater treatment operations: collecting and analyzing QC samples, operating filter press, monitoring process, and maintenance activities; material typically present in waste stream at less than 1%.

3. Provide industrial hygiene monitoring data, if available, with a brief description of the sampling method and exposure scenario monitored, e.g., describe the specific worker activities performed by the individuals monitored. For privacy considerations, please do not include any personal identifiers such as a worker's name or social security number with any data submitted to EPA.

Personal sampler tubes with XAD-4 resin and mixed cellulose ester or glass fiber prefilter. Sample analysis is by LC-MS. Previous sampling had employed silica gel acid mist tubes with GC-ECD analysis. See attached table for air sample results.

There has been area/source air monitoring data and/or surface wipe sampling data collected for this material at the plant. Area/source sample results and/or surface wipe sample results are used to identify areas with employee exposure potential as part of exposure assessment under 3M's industrial hygiene program and are not measurements of actual employee exposures. Hence, they are not included with this submission. Prior to 1999, these samples were considered to be semi-validated.

Surfaces in production and administration areas were sampled in 1998. A glass fiber filter wetted with methyl alcohol was wiped inside a 10 cm x 10 cm square with analysis by LC-MS. Results indicated the presence of these materials on floors and equipment surfaces in production areas. This resulted in improvements to Hazard Communication practices, personal hygiene emphasis, personal protective equipment emphasis, and several engineering and administrative changes.

The sample results of any air monitoring are compared to 3M's voluntary exposure guideline (EG) of 0.1 mg/m3 (milligrams of fluorochemical per cubic meter of air) for various fluorochemical compounds, including compounds covered in this submittal. The EG is an 8-hour time-weighted average (TWA) personal breathing zone exposure chosen to minimize potential for uptake.

Each 3M plant that produces sulfonated fluorochemicals has an industrial hygienist on staff and is supported by a corporate industrial hygiene group. 3M's industrial hygiene program focuses on task-based exposure assessment and control. Exposures are identified and assessed qualitatively and/or quantitatively. Qualitative assessments are performed by an industrial hygienist. Quantitative assessments include task-based personal sampling for certain, specific fluorochemicals and/or source or area sampling. The results of the assessments support decisions on exposure control. Engineering controls are preferred, but personal protective equipment may be used on an interim basis or when effective engineering control is not feasible.

Air Sampling Data for 25268-77-3

Job Classification	Sample number	Concentration (mg/m3)	Minimum (mg/m3)	Maximum (mg/m3)	Geometric Mean	Geometric Standard Deviation
		Totals →	<0.003	0.46	0.025	4.6
Chemical	90-080	0.12				
Operator	92-060	0.06				
Building 3	92-061	0.27				
	94-031*	<0.01				
	94-032*	<0.01				
	94-034*	<0.01				
	94-037*	<0.01				
	94-038*	<0.01				
	94-039*	<0.006				
	94-041*	<0.01				
	94-046*	<0.01				
	94-050	<0.01				
	94-075	<0.006				
	94-078	<0.008				
	95-006*	0.012				
	95-007*	0.007				
	95-014*	0.27				
	95-040*	0.46				
	95-041*	0.019				
Chemical Operator	95-024*	0.28				
Flaker	97-091*	0.15				
Team Leader	92-060	0.06				
Building 3	92-061	0.27				
	94-036*	<0.003				
	94-047*	<0.02				
	94-051*	<0.005				
	94-054*	<0.005				
	94-055*	<0.005				
	96-069	0.16				
	98-481*	0.05				
Team Leader Flaker	98-098*	0.049				

^{*} The value for this sample is one of several fluorochemical analytes collected on the sample. Samples are all personal breathing zone samples taken over the duration of a specific task.

4. Briefly describe the engineering controls used to minimize exposure to this chemical:

Materials are transferred using closed piping (where possible) from reactor vessels to other containers. Vacuum charging of materials from drums is a standard practice. Local exhaust ventilation hoods are situated at significant point sources such as at drum bungs when drumming. Some equipment, such as the flaker, is equipped with local exhaust ventilation. General room air provides for dilution of airborne materials.

5. Briefly list the personal protective equipment your workers regularly wear to prevent exposure of this chemical:

Specific personal protective equipment is indicated on written operating procedures used by process operators.

Respiratory protection: Tasks with significant exposure potential (charging, drumming, some maintenance tasks) are performed using positive pressure, pressure demand full face supplied air respirators. Tasks with moderate exposure potential (short term sampling from taps or drums, and some maintenance tasks) are performed using full face air-purifying respirators with organic vapor cartridges and P100 prefilters.

Hand protection: All tasks identified as having potential for hand contact require neoprene (Scorpio) or nitrile industrial gloves.

Body protection: Exposure significant tasks use one or two-piece PVC suits. Aprons may be worn for some low exposure potential tasks such as sampling.

Foot protection: Tasks identified as posing a significant foot exposure potential require chemical resistant boots.

Comments: (This section is available to clarify the responses given. Attach additional pages if desired.)

Current exposures are expected to be less than those measured in the recent past. Significant sources have been identified and eliminated. In 1998 an extensive area air sampling of the production areas was performed. Areas with higher levels of airborne materials were investigated further to identify sources. These sources, which include the flaking equipment, are in the process of being eliminated or reduced through replacement with better ventilated equipment. In addition, the ventilation system in the primary production building (3) is being upgraded.

The fluorochemical hazard communication program was improved upon in 1998. This has resulted in improved exposure avoidance behaviors among production employees and better use of with personal protective equipment requirements.

Additional hand wash facilities have been installed in primary process buildings. Food consumption and other hand-to-mouth activities are prohibited. There is no smoking in process buildings. Process employees are instructed to remove and leave their work boots and work coveralls at the plant. Work coveralls are professionally laundered.

Non-production equipment surfaces such as floors, doorknobs and stair rails are cleaned frequently.

VII. CHEMICAL END USES

A. END USE AS AN INTERMEDIATE CONSUMED TO MAKE OTHER CHEMICALS

A1. On-Site Use as a Intermediate:

Intermediate consumed in the production of several polymeric products which are used as protective treatments for circuit boards, nonwovens, apparel and leather.

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Fluoroaliphatic Acrylates

90-95%

2. Fluorochemical Acrylates CAS 92265-81-1

3-8%

As reported in Part III, p.2

A2. Off-Site Use as an Intermediate

Product chemical class or product chemical (Include CAS number if appropriate) % of total volume of subject chemical manufactured or imported

1. Not applicable.

As reported in Part III, p.2

B. END USES OTHER THAN AS A CONSUMED INTERMEDIATE

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Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be mill applied as protective, repellent treatments for apparel substrates. These substrates will be made into apparel products to be used by consumers and commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported Volume going to this use: 1-2	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)
Use Number 2 of 8 Description of Chemical End Use: This CAS number and the industrial use repellent treatments for carpeting. The carpeting windustry. This CAS number itself will be present in	ers to be mill applied as protective, vill be used by consumers and commercial
Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: _X <1% (<5000 ppm) 1-30% 30-60% 60-90% >90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)

Use Number 3 of 8

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are sold to industrial users to be mill applied as protective, repellent treatments for nonwoven substrates. These substrates will be made into industrial workwear or medical garments to be used by commercial industry. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: \[\frac{X}{2} & < 1\% \\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vaporX Liquid solution Other (Explain)
Use Number 4 of 8 Description of Chemical End Use: This CAS manufacture polymers that are sold to industrial repellent treatments for upholstery. The upholst commercial industry. This CAS number itself was a residual.	users to be mill applied as protective, tery will be used by consumers and
Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate Weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid
X <1% — 1-30% — 30-60% — 60-90% — >90%	Water or solvent – wet solid Gas or vapor X Liquid solution Other (Explain)

Use Number 5 of 8

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture materials that are regulated by the Food and Drug Administration as indirect food additives used to impart grease and oil resistance to paper and paperboard. Paper or paperboard are used in food packaging products such as bags, wrappers, cartons, or trays. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)
Use Number 6 of 8 Description of Chemical End Use: This CAS number manufacture materials that are sold to industrial use treatments for carbonless paper (business forms). These products at low levels as a residual.	rs to be mill applied as protective
Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:
If used in a mixture check appropriate box to indicate Weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)

Use Number 7 of 8

Description of Chemical End Use: This CAS number is used as an intermediate to manufacture materials that are sold to industrial users to be applied as protective treatments for porous surfaces. This CAS number itself will be present in these products at low levels as a residual.

Percent of total manufactured or imported volume going to this use: <1 ±	Check all physical forms of the chemical during this use:	
If used in a mixture check appropriate box to indicate weight fraction. Average values are acceptable: <1% 1-30% (0.1-2%) 30-60% 60-90% >90%	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid Gas or vapor Liquid solution Other (Explain)	
Use Number 8 of 8 Description of Chemical End Use: This CAS number is used as an intermediate to manufacture polymers that are incorporated into products for consumer use as protective, repellent treatments for upholstery. This CAS number itself will be present in these products at low levels as a residual.		
Percent of total manufactured or imported volume going to this use: <1 \pm	Check all physical forms of the chemical during this use:	
If used in a mixture check appropriate box to indicate Weight fraction. Average values are acceptable:	Aerosol Dry Powder Pellets or large crystals Water or solvent – wet solid	
X <1% 1-30% 30-60% 60-90% >90% >90%	Gas or vapor Liquid solution Other (Explain)	